



Research Report

***Laboratory Testing of
Crack Sealing Materials
for Flexible Pavements***

***Transportation Association of Canada
Association des transports du Canada***

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Abstract <p>The aim of this project was to develop and/or identify an effective laboratory test method and equipment design for prediction of field performance of crack sealants. The specific objectives were to 1) identify factors critical for the field performance of crack sealants; 2) select and develop a test method that reflects these factors; 3) design testing instruments capable of performing the selected performance test; and 4) verify the laboratory test methods by comparing test results to known field performance of crack sealants.</p> <p>The project was conducted in three phases: a) a survey of crack sealing practices in Canada and a literature review of the factors influencing the field performance of crack sealers and available test methods; b) the development of a laboratory test method to measure the parameters most critical to field performance, and c) verification of the test methods by comparison of test results with known field performance data.</p> <p>Since the behaviour of crack sealants at low service temperatures influence field performance, two tests reflecting this behaviour were identified and designed. They are the Low Temperature Stress Relaxation Test and the Tensile Adhesion Test. Fourteen crack sealants with known field performances were evaluated by these two methods.</p> <p>It was concluded that if the crack sealant fails either the Low Temperature Stress Relaxation Test or the Tensile Adhesion Test, the probability of failure in the field increases. If the crack sealant fails both tests, it is not recommended for use in the field. A detailed protocol for both test methods is presented in the report.</p> <p>It is recommended that the findings of this report be verified, in different laboratories, on a greater number of crack sealants with known field performance. If the project conclusions are confirmed, the test methods should be used in the CBSB (Canadian General Standards Board) crack sealant specification. At the same time, the testing methods without relationship to crack sealant field performance should be removed from the specification.</p>		Keywords (IRRD) Joint Sealing 3608 Cracking 5211 Flexible Pavement 2944 Canada 8018	
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Résumé <p>Le présent projet avait comme but d'établir une méthode d'essai de laboratoire permettant de déterminer le rendement in situ des scellants à fissures. Les objectifs précis du projet étaient 1) de préciser les aspects critiques du rendement in situ des scellants à fissures; 2) de choisir et de parfaire une méthode d'essai axée sur ces aspects critiques; 3) de mettre au point des instruments adaptés à ce genre d'essai; et 4) de vérifier les méthodes d'essai de laboratoire en comparant les résultats à des observations du rendement réel des scellants à fissures.</p> <p>Le projet a comporté trois étapes : 1) un sondage des méthodes de rebouchage utilisées au Canada et une synthèse documentaire des paramètres qui influencent le rendement des scellants à fissures d'une part et des méthodes d'essai disponibles d'autre part; 2) l'établissement d'une méthode d'essai de laboratoire capable de mesurer les principaux paramètres de rendement et 3) la vérification des méthodes d'essai par comparaison des résultats à des données réelles de rendement.</p> <p>Compte tenu de l'effet sur le rendement des scellants à fissures de leur comportement à basse température, on a établi deux essais se rapportant à ce type de comportement. Il s'agit de l'essai de relaxation à basse température et de l'essai d'adhérence en traction. On a évalué 14 scellants à fissures dont le rendement était connu à l'aide de ces deux méthodes.</p> <p>On a conclu que la probabilité de rupture in situ augmente si le fonctionnement du scellant à fissures n'est pas satisfaisant soit pour l'essai de relaxation à basse température, soit pour l'essai d'adhérence en traction. Un produit qui donne des résultats insatisfaisants pour les deux essais n'est pas recommandé. Le rapport présente un protocole détaillé pour les deux méthodes d'essai.</p> <p>Il est recommandé que l'on vérifie les résultats de ce rapport, dans des laboratoires différents, sur un plus grand nombre de scellants à fissures à rendement connu. Si les conclusions du projet sont confirmées, les méthodes d'essai devraient être intégrées à la norme de l'ONGC (Office des normes générales du Canada) qui se rapporte aux scellants à fissures. De plus, les méthodes d'essai qui ne rapportent pas au rendement des scellants à fissures in situ devraient être éliminées de la norme.</p>			Mots-clés Remplissage des joints 3608 Fissuration 5211 Chaussée souple 2944 Canada 8018
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The first phase of this project dealing with the literature survey of crack sealing was led by Mr. R. David Watson and is described in the progress report "TRANSPORTATION ASSOCIATION OF CANADA RESEARCH PROJECT 92-31 - LITERATURE SURVEY OF CRACK SEALING" authored by Mr. R. David Watson and issued in December 1993.

The second phase of this project dealing with laboratory testing procedures and instrument design was led by Mr. R. David Watson and is described in the progress report "TRANSPORTATION ASSOCIATION OF CANADA RESEARCH PROJECT 92-31 - PROGRESS REPORT ON TASKS 3 & 4 LABORATORY TESTING PROCEDURES & INSTRUMENT DESIGN" authored by Mr. R. David Watson and Mr. Dale Sieben and issued in January 1995.

The rheological analysis of tests developed in this project was performed in cooperation with Dr. Jiri Stastna.

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EXECUTIVE SUMMARY

The use of hot pour crack sealant for the maintenance of asphalt concrete pavements in Canada is a common practice. Crack sealants decrease the amount of road maintenance required and extend the life of pavement before major reconstruction is necessary. However many crack sealants, even those described as high performance crack sealants, last only for a short period of time and cracks in pavements need frequent resealing.

One of the reasons that poorly performing crack sealants are in use is the lack of laboratory tests which predict the field performance of these materials. The aim of this project was to develop and/or identify an effective laboratory test method and equipment design for prediction of field performance of crack sealants.

The specific project objectives were:

- to identify factors critical for the field performance of crack sealants;
- to select and develop a test method that reflects these critical factors;
- to design testing instruments capable of performing the selected performance test;
- to verify the laboratory test methods by comparing test results to known field performance of crack sealants.

The project was conducted in three phases: 1) a survey of crack sealing practices in Canada and a literature review of the factors influencing the field performance of crack sealers and available test methods; 2) the development of a laboratory test method to measure the parameters most critical to field performance, and 3) verification of the test methods by comparison of test results with known field performance data.

Factors which influence the performance of crack sealants in the field can be divided into two groups: a) those which are a function of crack sealant properties (e.g. rheological properties of the sealant - fluidity, softness, hardness or bond strength toward the crack wall) and b) those which are not a function of crack sealant properties (e.g. quality of installation, type of winter maintenance). Only the first group can be addressed in any laboratory testing methods.

The testing methods found in different existing crack sealant specifications are mostly identical. Some play important roles in determining necessary properties of crack sealants (e.g. flow at high service temperature, stability and compatibility tests). Others are of dubious value, poor reproducibility, or both (e.g. bond test, resilience test).

Since the behaviour of crack sealants at low service temperatures influence field performance, two tests reflecting this behaviour were identified and designed.

First, a Low Temperature Stress Relaxation Test measures the resistance of crack sealant to extension at the speed of 1 mm/min and the capability of the material to relax the cumulated stresses after a defined level of extension is achieved. The idea behind the test is that during the widening of the crack at low service temperatures, the crack sealant must retain the ability to change shape and also dissipate

the imposed stresses as quickly and as much as possible. This behaviour will minimize the stresses imposed on the bond between the crack sealant and the crack wall.

Second, the Tensile Adhesion Test measures the resistance of the crack sealant to debond from a solid surface. This test is performed at the speed of 10 mm/min to minimize the relaxation, which may occur during the extension period.

Both tests can be performed on any tensile testing apparatus capable of generating tensile stress of 2000N, and with the ability to pull the sample at two constant speeds (1 mm/min and 10 mm/min) and with the ability to monitor the change of tensile stress. The apparatus has to be equipped with an environmental chamber capable of accurately maintaining a temperature of -30°C. The forms for tests are inexpensive and easy to manufacture.

The Testing Protocol was developed using 16 commercially manufactured crack sealants. Their field performance was not known.

In the next phase of the project, 14 crack sealants with known field performances (only field performances of 12 crack sealants were reported) were evaluated by the two previously mentioned tests. Of these, eight performed satisfactorily and four failed.

Four criteria, two from each test, appear to be related to the field performance of the crack sealants:

Cold Temperature Stress Relaxation Test

- peak load of the crack sealant
- tendency of the crack sealant to break during the extension period.

Of the 12 crack sealants with known service performances, five failed this test. All four crack sealants that failed in the field were among these five.

Tensile Adhesion Test

- extension of the crack sealant in moment of complete debonding
- work necessary for the total crack sealant debonding.

Of the 12 crack sealants with known service performances, four failed this test. These were the same four that failed in the field.

It was concluded that if the crack sealant fails either the Cold Stress Relaxation Test or the Tensile Adhesion Test, the probability of failure in the field increases. If the crack sealant fails both tests, it is not recommended for use in the field. A detailed protocol for both test methods is presented in the report.

It is recommended to verify the findings of this research project on the larger number of crack sealants with known field performance, and in different laboratories. If the project conclusions are confirmed, the test methods should be implemented into the CGSB crack sealant specification. At the same time, the testing methods without relationship to crack sealant field performance should be removed from the specification.

SOMMAIRE

Le recours à un scellant à fissures coulé à chaud pour l'entretien des chaussées de béton bitumineux est chose courante au Canada. Les scellants à fissures permettent de réduire l'envergure des travaux d'entretien routier et de prolonger la vie des chaussées. Toutefois, de nombreux scellants à fissures, y compris des produits à haute performance, ne sont pas très durables, de sorte qu'il faut prévoir des travaux de rebouchage fréquents.

Une des raisons qui expliquent le rendement médiocre des scellants à fissures est l'absence d'essais de laboratoire capables de déterminer le rendement in situ de ce genre de produit. Le présent projet avait comme but d'établir une méthode d'essai de laboratoire permettant de déterminer le rendement in situ des scellants à fissures.

Les objectifs précis du projet étaient les suivants :

- préciser les aspects critiques du rendement in situ des scellants à fissures;
- choisir et parfaire une méthode d'essai axée sur ces aspects critiques;
- mettre au point des instruments adaptés à ce genre d'essai;
- vérifier les méthodes d'essai en laboratoire en comparant les résultats à des observations du rendement réel des scellants à fissures.

Le projet a comporté trois étapes : 1) un sondage des méthodes de rebouchage utilisées au Canada et une synthèse documentaire des paramètres qui influencent le rendement des scellants à fissures d'une part et des méthodes d'essai disponibles d'autre part; 2) l'établissement d'une méthode d'essai de laboratoire capable de mesurer les principaux paramètres de rendement et 3) la vérification des méthodes d'essai par comparaison des résultats à des données réelles de rendement.

Les paramètres qui influencent le rendement des scellants à fissures sont de deux types : ceux qui dépendent des propriétés du produit (p. ex. propriétés rhéologiques, fluidité, souplesse, rigidité ou résistance à l'arrachement) et ceux qui ne dépendent pas des propriétés du produit (p. ex. qualité des travaux de mise en place, type d'entretien en hiver). Seul le premier type peut faire l'objet d'essais de laboratoire.

Les méthodes d'essai que l'on trouve dans les principales normes se rapportant aux scellants à fissures sont à peu près identiques. Les unes jouent un rôle important dans la détermination des propriétés requises (p. ex. écoulement à des températures de service élevées, stabilité, compatibilité); les autres sont de qualité douteuse ou ne sont pas facilement reproductibles (p. ex. essai d'adhérence, essai de résilience).

Compte tenu de l'effet sur le rendement des scellants à fissures du comportement à basse température, on a établi deux essais se rapportant à ce type de comportement.

Le premier, un essai de relaxation à basse température, mesure la résistance du scellant à fissures à un étirement de 1 mm/min, ainsi que l'aptitude du produit à permettre un relâchement des contraintes accumulées après un certain degré d'étirement. Compte tenu de l'élargissement des fentes à de basses températures de service, le scellant à fissures doit pouvoir changer de forme tout en assurant une détente des contraintes qui soit rapide et optimale. Un tel comportement réduit au minimum les contraintes qui surviennent à l'interface entre le produit et la paroi des fentes.

Le deuxième, un essai d'adhérence en traction, mesure la résistance du scellant à fissures au décollement sur une surface solide. Cet essai se déroule à une vitesse de 10 mm/min de façon à minimiser la relaxation qui risque de se produire durant l'étirement.

Ces deux essais peuvent être menés à l'aide de tout appareil capable de produire des contraintes de traction de 2 000N et d'étirer des échantillons à deux vitesses constantes, soit 1 mm/min et 10 mm/min, l'évolution des contraintes étant enregistrée. L'appareil doit être accompagné d'une enceinte expérimentale capable de maintenir la température à exactement -30 °C. L'outillage des essais est bon marché et facile à fabriquer.

Le protocole d'essai a été mis au point à l'aide de 16 scellants à fissures que l'on trouve sur le marché. Leur rendement in situ était inconnu.

Durant l'étape suivante du projet, 14 scellants à fissures dont le rendement était connu (le rendement de 12 produits seulement a été signalé) ont été évalués à l'aide des deux essais mentionnés. Huit des 12 produits ont donné des résultats satisfaisants.

Quatre critères, c'est-à-dire deux pour chaque essai, semblent se rapporter au rendement in situ des scellants à fissures :

Essai de relaxation à basse température

- charge maximale,
- tendance du scellant à fissures à la rupture durant l'étirement.

Cinq des 12 scellants à fissures à rendement connu ont donné des résultats insatisfaisants. Les quatre scellants à fissures qui n'ont pas bien fonctionné in situ figureraient parmi ces cinq produits.

Essai d'adhérence en traction

- étirement du scellant à fissures au moment du décollement complet,
- effort requis pour le décollement complet du scellant à fissures.

Quatre des 12 scellants à fissures à rendement connu ont donné des résultats insatisfaisants. Il s'agissait des quatre mêmes produits qui n'avaient pas bien fonctionné in situ.

On a conclu que la probabilité de rupture in situ augmente si le fonctionnement du scellant à fissures n'est pas satisfaisant soit pour l'essai de relaxation à basse température, soit pour l'essai d'adhérence en traction. Un produit qui donne des résultats insatisfaisants pour les deux essais n'est pas recommandé. Le rapport présente un protocole détaillé pour les deux méthodes d'essai.

Il est recommandé que l'on vérifie le résultat de ce programme de recherche sur un plus grand nombre de scellants à fissures à rendement connu dans des laboratoires différents. Si les conclusions sont confirmées, les méthodes d'essai devraient être intégrées à la norme de l'ONGC qui se rapporte aux scellants à fissures. De plus, les méthodes d'essai qui ne se rapportent pas au rendement des scellants à fissures in situ devraient être éliminées de la norme.

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APPENDIX: Rheological Analysis of the Stress Relaxation Test and Behaviour of Crack Sealants

1.0 INTRODUCTION

The use of hot pour crack sealants in Canada for highway maintenance is a common practice, costing several million dollars per year. Increased demands for better performance of pavements and limited government budgets have placed more stress on the transportation system. Escalating traffic volumes and heavier loads have heightened the need for superior performance in both pavements and crack sealants. Crack sealants that perform for longer periods of time decrease the amount of road maintenance required and extend the life of the pavement before major reconstruction is necessary.

Specifications and laboratory testing procedures now in place do not adequately describe the properties of crack sealants to predict their road performance. Some of the tests did not give reproducible results, for example, the bond or resilience test. Field conditions need to be better reflected in the laboratory tests. At the same time, the practicality of such tests has to be maintained.

At the present time there are a number of sealants containing polymers, especially rubber modified asphalt, on the market with newer products appearing each year. The performance of these new materials is difficult to predict without using lengthy and expensive field testing. Even the results from field trials are sometimes ambiguous because of the unique circumstances and conditions existing at each field test project. Many times the results show good performance at one site and poor performance at another.

The thrust of this project, therefore, was to develop and/or to identify an effective test method and equipment design to evaluate crack sealants so their performance in the field could be predicted. The comparison of results of the selected test methods with the field performance of several commercially manufactured materials was also part of the project.

2.0 PROJECT OBJECTIVES

The central objective of the project was to develop an effective test method and apparatus design to evaluate crack sealing materials for maintaining asphalt pavements. Part of the project was to address the correlation of laboratory test data with field performance of the materials.

The specific objectives were:

1. The identification and quantification of the factors critical to the field performance of crack sealants under conditions prevailing in Canada.
2. The selection and development of the test method that reflects the factors and conditions that were determined to affect crack sealant performance.
3. The design of a testing instrument capable to test the performance of crack sealing materials. The testing and evaluating process should result in more reliable feedback of expected field performance.
4. To compare the laboratory test parameters with the field performance data.

3.0 RESEARCH PLAN

1. Literature and technology review of crack sealing

Critical literature review on latest crack sealing technology available and the review of the crack sealing testing methods through the use of the extensive on-line capabilities of the Novacor Research and Technology Corporation (NRTC) library.

2. Identification of field performance parameters for crack sealing

Examination of parameters affecting field performance of crack sealants. Factors like crack preparation, application methods, climatic conditions and road maintenance during winter months are of special attention.

3. Development of laboratory testing procedure

Evaluation of potential methods to measure the capability of crack sealant to maintain a bond to the crack wall under adverse conditions. Selection of the most promising method. Development of the test.

4. Identification of quality assurance tests.

Identification of auxiliary tests which affect the performance, application, and durability of the crack sealant.

5. Design of testing equipment

The design of testing equipment, test procedure for evaluating the bond strength or other important crack sealant properties at low service temperatures.

6. Development of testing protocol

Establishment of testing protocol for the newly developed test.

7. Evaluation of commercially available crack sealants

Collection of several commercially available crack sealants with known field performance. Evaluation of collected crack sealants using the newly developed test. Comparison of laboratory test data with the field performance of crack sealants.

8. Final report

A final report summarizing all works performed within the project would be prepared.

**Literature Review on
Crack Sealing and
Testing of Crack Sealants**

4.1 INTRODUCTION

The literature research was carried out to review the available information on factors influencing the performance of crack sealants and available testing methods. The literature research was performed through Novacor Research & Technology Corporation databases and the most pertinent papers selected.

4.2 SPECIFICATIONS

There are specifications in the U.S.A. and Canada that describe the test procedures and required properties of crack sealants. The most commonly used specifications are ASTM D3405, ASTM D3407, ASTM D1190, U.S. Federal Specification SS-S-1401C, AASHTO T187-60, and the related Canadian Specification CGSB-37.50-M89. These specifications are very similar, quoting many of the same test procedures and property ranges. There are also numerous local agency specifications that apply to the local conditions which are based on the previously mentioned national specifications. The main specifications were listed in the Progress Report "Literature Survey of Crack Sealing", issued in December 1993.

These specifications deal with testing of crack sealants to mainly ensure that sealant properties are consistent, but the tests do not really relate to the crack sealants' performance in the field under real conditions. Sealants can comply with these specifications and have similar test results but experience has shown they behave very differently when placed in the field. This theme of unpredictability reoccurs in many research papers over the past 30 years. It can be concluded that the specifications are not addressing the proper parameters or combination of parameters to assess and predict the field performance of crack sealants.

4.3 LABORATORY TEST METHODS

Laboratory test methods are basically the same throughout the national standards of ASTM, AASHTO, U.S. Federal, and CGSB. However, in the literature over the past 30 years, there have been several attempts to develop alternative tests to those in the specifications, particularly the bond test. A discussion of the tests used in specifications and modified test procedures is provided in the following sections.

Conventional Methods

The methods discussed below are part of AASHTO, T187-60, ASTM D3407 and other specifications.

Cone Penetration at 25 C

Cone penetration is a measure of consistency of the material and has no significant value in predicting performance. The maximum specified limit of 90 dmm may impose worsening of the low temperature behaviour of the crack material because it limits material softness. The softness of crack sealant is more effectively monitored by the flow at 60 C. Cone penetration test is more or less redundant.

Flow at 60 C

The flow test is important from a performance perspective to ensure the material stays in the crack at the highest temperatures experienced during the summer. The specified value is 3 mm. This requirement may be too severe. Surface oxidation of the crack sealant inhibits flow very early after placement and generally flow has not been seen as a problem in the field.

Resilience at 25 C

The resilience test by virtue of its design may eliminate crack sealants that perform very well in the field. This test measures the rebound of the crack sealant, requiring a 60% recovery in 20 seconds. The demand to have a material which recovers very quickly requires formulations of crack sealants with high amounts of rubbery polymers. The cohesive strength in produced crack sealants may be too high. A balance needs to be achieved between the resilience and the ductile flow of the sealant especially at cold temperatures.

Flexibility at -25 C

The low temperature flexibility test gives some indication of sealant brittleness but relates little to its actual performance. This test may be helpful if the material is placed on a roof.

Bond Test

The bond test is performed under two sets of conditions, either at -29°C for 3 cycles, non-immersed and immersed or at -18°C for 5 cycles. Specimens are examined after each extension for cracks or separations. This test has created controversy for the past 30 years. The bond test is considered very unreliable and has poor repeatability. Many authors have questioned its validity because materials that passed the bond test in the laboratory showed very poor results when applied in the field and vice versa.

Stability and Compatibility Test

The heat stability tests and compatibility tests of crack sealant with asphalt pavements are very useful. They predict potential problems with product instability after prolonged heating during application and incompatibility between the asphalt mix and crack sealant, which may cause poor bond between them.

Modified Methods

The bond test has been the most controversial test over the years and modifications were proposed without subsequent success in changing the specifications. The bond test shows a history of inconsistency of test results which lead to questions about its validity, the manner in which the test is performed, and interpretation of test results. E. Tons from MIT carried out a study in 1959 in which sixteen laboratories tested the same crack sealant using bond test, cone penetration and flow test (1). The study revealed significant variation in the bond test results between laboratories. Nevertheless, this test continues to be used in crack sealant specifications. The test in its present form yields little useful information regarding the performance of crack sealant under field conditions.

In the late 1950's, W.H. Kuenning of the Portland Cements Association evaluated a number of different sealants using a different type of bond test (2). The sealants were placed between concrete blocks (62.5 x 100 x 400 mm) with a 50 mm deep joint (saw-cut) at the half-way point with the remainder of the crack created by breaking by hand the remaining concrete below the cut joint. The sealant was extended at a rate of 0.78 mm per hour to 100% extension at -18°C. The crack sealants were also cycled at 23°C using the same rate of extension, starting with maximum extension of 60% and increasing the maximum extension by 20% each additional cycle to an extension of 160%. Kuenning also evaluated the effect of the shape factor on the performance of joint sealants and found some correlation between the dimensions of the cross-section of the crack sealant and its performance. He found some sealants performed well under severe conditions but laboratory test results were never compared with the crack sealants' field performance.

Another test was designed by R.A. Jimenez and reported in 1988. This test used crack sealant beams 300 x 125 x 75 mm (3). These beams were subjected to continuous slow extension at 25°C and 0°C respectively until failure. Sealants that passed were tested using a fast repetitive rate of extension and compression at 25°C and 0°C respectively with three different values of deformation. Jimenez found that the slow extension and the fast extension-compression at 25°C gave consistent results but the cycle of fast extension-compression at 0°C needed modification. The data were inconsistent because of limited stiffness in the apparatus. One significant drawback of this test apparatus was that it required a walk-in freezer unit to perform the testing. Also, 0°C is not low enough to address the performance of the sealant at low field temperatures.

These are only a few of the attempts made to develop more reliable bond test. There are many more variations of the bond test listed in the literature but all have a similar theme. The results obtained from the modification of the bond test were inconclusive when correlated with the field performance. Some studies aimed at correlation of the field performance and results from laboratory tests by the U.S. Army Corps of Engineers are still ongoing.

4.4 CRACK PREPARATION TECHNIQUES

The general consensus in crack preparation for the purpose of sealing is to ensure that cracks are properly routed and dry. The cracks should then be cleared of dust and debris by use of at least compressed air or preferably hot compressed air and lanced. These precautions ensure that the best possible bond between crack sealant and crack wall will be achieved.

When using hot poured sealants to fill cracks, opinions vary slightly as to how wide and how deep to rout the cracks. Some suggest the cracks should be routed to a minimum of 15 mm in width. The routing should be done under dry conditions for best results to prevent the formation of a dust and water slurry. This type of slurry is difficult to remove using only backpack blowers or compressed air and would require the use of a hot air compressor. L.N. Lynch (9) suggests that a crack 6 to 12 mm wide should be widened to 15 mm and any random crack wider than 19 mm should only be sandblasted and cleaned. The routed cracks should be sandblasted on the same day as they are sealed and all debris should be removed from the crack and the upper surface of the pavement on both sides of the crack to ensure a proper bond of the sealant. Also complete removal of any previous joint sealing material is necessary to promote the best possible bond of the crack sealant to the crack wall.

4.5 CRACK FILLING TECHNOLOGY

The technology for filling cracks varies from pump-fed wands, gravity cones, to heated gravity fed systems. All of the systems have some advantages and disadvantages but as long as the crack sealant is at pouring temperature all these systems work. L.N. Lynch (9) suggests that crack sealants should not be gravity fed into cracks but extruded to prevent bubbling of the surface and the nozzle should fit inside the crack. This would be the ideal situation if the equipment were always available.

A number of factors besides the equipment used to fill the cracks can affect the performance of the crack sealant. In our experience, the temperature at which the crack sealant is poured is critical to develop the bond to the crack wall. Another factor which may affect the performance is the shape factor or width to depth ratio of the routed crack. Some publications indicate the shape factor has a significant impact on performance where others claimed the shape factor has no effect at all. Dust and moisture present on the crack surfaces is also a factor that may severely inhibit the formation of a good bond between the crack surface and the sealant. It is also suggested in the literature that crack sealants must have some ability to bond to dusty or moist surfaces or both. The dust and moisture are a fact of life and may be addressed by maintaining recommended pouring temperatures and placing some upper limit on the viscosity of the joint sealant.

4.6 FIELD EVALUATION TECHNIQUE

Generally, most failures of sealed cracks in Canada tend to be adhesion failures during the cold winter months. They are caused by a weak bond between the crack wall and the sealant and are supported by the hardness and the high internal cohesive strength of the crack sealant. This is compounded by other factors, such as poor workmanship during application, use of salt and sand, use of snowplows, and the hydraulic action of water. The assessment of crack sealant failure is still very much a visual inspection, which still remains the best way of determining the success of a job after placement of the crack sealant. Some of the most recent studies on the use of crack sealants in Canada were done by J.F. Masson *et al* (5,6).

4.7 IMPORTANT PARAMETERS OF CRACK SEALANT PERFORMANCE

As mentioned before, most failures of crack sealants in Canada tend to be adhesion failures, caused by weak bonding and by the inability of the sealant to change its shape and to dissipate the stresses built up during the stretching when the temperature decreases.

The internal cohesive strength, sometimes fostered by specifications like ASTM D-3405, reduces the capability of the material for ductile flow and for dissipation of cumulated energy. The tension built in the sealant surpasses the bond strength. The gain in resilience and the existing upper limit in cone penetration may actually prefer the harder materials with limited capability to flow. No joint sealant has the strength to hold a road together, therefore, the crack sealant must have the ability to adjust its shape and to dissipate energy produced during this adjustment.

In addition to cold weather performance of the crack sealant, the quality of workmanship during routing of cracks and the application of the crack sealants is very important for future crack sealant performance. Preparation of the crack and application of the sealant are critical in promoting a good bond between the crack sealant and the crack wall. If attention is not paid to workmanship, the best crack sealant performance possible will not overcome the consequences of poor installation.

More details on the literature review may be found in the progress report "Transportation Association of Canada Research Project 92-31 - Literature Survey of Crack Sealing" from December 1993, authored by R. David Watson.

4.8 LITERATURE SUMMARY

The following is a summary of the most pertinent papers on the subject of crack sealing.

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17. Investigations into Improving Field-Molded Pavement Joint Sealant Field Performance", Concrete International, pp.57-60, June 1992.
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**Development of the Test Methods
for Characterization of Crack Sealants
and Design of the Testing Equipment**

5.1 INTRODUCTION

As already discussed in the chapter on the literature survey, the prediction of crack sealant performance in sealing the asphalt concrete pavements is very difficult to achieve with any reliability. There are multiple variables that affect the functionality of the crack sealant to allow prediction of its field performance, especially from laboratory generated data, but even from the field trials.

The parameters which decide the performance of the crack sealant can be divided into two groups, subjective and objective.

The subjective parameters identify the characteristics of the crack sealant itself. These parameters should describe:

- the ability of the sealant to be heated to application temperature and be maintained at that temperature for the necessary time without the separation of the material or undue changes of its properties;
- the fluidity of the sealant at the application temperature to be poured into the crack, to enter the crevices and pores of the crack wall and to fully adhere to the crack walls. On the other hand, the fluidity of the sealant must not be such that the sealant would flow out of the crack;
- the ability of the sealant to withstand the high service temperature without becoming too soft, so that the sealant would not be removed from the crack by the ongoing traffic;
- the ability of the sealant to withstand the low service temperatures without becoming too hard. It has to be able to expand with the crack and to release stresses caused by the expansion;
- the strength of the bond between the crack wall and the sealant. This strength should be larger than the maximum built-up stress in the sealant.

The objective parameters identify the conditions of the sealant installation and the conditions at which the sealant will have to perform. These parameters can be predicted or regulated to a different degree. These parameters are:

- technology of the sealant installation;
- size and shape of the crack;
- chemical composition of the aggregate in the crack wall and the porosity of the wall;
- weather conditions (minimum and maximum service temperature, freeze-thaw cycles, amount of rain, snow and ice);
- water table level;
- traffic density;
- winter pavement maintenance.

The tests and specifications of sealants can reflect some of these parameters, especially those more closely related to the physical properties of the sealant. Tests and specifications can, to a degree, reflect other parameters such as weather conditions, but cannot reflect others, e.g. the underground water pressure.

Some parameters, which cannot be captured by the tests and specifications, can be influenced by the technological guidelines (size and shape of the crack, technology of the sealant installation); others will remain a wild card and will be left to the judgment and experience of a local highway engineer.

5.2 EVALUATION METHODS FOR CRACK SEALANT

Properties at Medium and High Service Temperatures

When the existing specifications and testing methods are critically assessed, it appears that the properties of crack sealants at high and medium service temperatures are adequately covered except for the need of more accurate determination of the fluidity at installation temperature.

Fluidity at Installation Temperatures

The determination of fluidity at installation temperatures is left exclusively to the judgment of the installation crew. It is therefore necessary to more exactly determine the viscosity at these temperatures. The development of the testing method is not necessary. The viscosity can be determined by any existing viscometer, capable of measuring viscosity of this material in this temperature region. The most suitable type of viscometer is any commercially available rotational viscometer capable of measuring the viscosity of materials up to 200°C. The proposed viscosity limits of crack sealant at installation temperatures will be discussed later.

Properties at Low Service Temperatures

Bond and Stress Relaxation at Low Service Temperatures

The low temperature behaviour of the crack sealant is presently determined by the bond test, and is generally reported as difficult to perform, very elaborate and with questionable reproducibility. The development of the method, or methods, to evaluate the performance of the crack sealant at low service temperature became the main focus of this project.

It is widely accepted that the majority of crack sealant failures occur when the crack sealant is exposed to the low service temperatures. At these temperatures the crack widens due to pavement shrinkage. At the same time, the crack sealant is hardest, least capable to adjust its shape to the crack opening. The expansion of the crack sealant causes a cumulation of stress in the material. If the cohesive forces in the crack sealant are larger than the adhesive forces between the crack sealant and the wall of the crack, and if the stress within the crack sealant becomes larger than the bond strength, the crack sealant will debond. If the bond strength is larger than the cohesive forces of the crack sealant and the stress becomes larger than the cohesive forces, the failure would occur within the crack sealant. Greater progression of the

crack opening and reduced capability of the crack sealant to relax the stress build-up will cause higher stress to build within the crack sealant and increased requirement on the bond strength.

From this analysis it was concluded that for the prediction of the performance of the crack sealant at low service temperatures, two types of information will be necessary: one describing the bond strength between the crack sealant and solid material, simulating the crack wall and, the second describing the build-up of the stress and capability of the crack sealant to relax this stress quickly.

After experimentation with the geometry of the tested samples, test conditions and other parameters, two tests, one for the evaluation of the stresses built during the crack sealant extension and the speed of their relaxation and the second, for the evaluation of the bond strength, both at low service temperatures were developed.

The design of the tests, the test equipment and the testing protocol are described below.

5.3 PROTOCOL FOR TESTING CRACK SEALANTS BY LOW TEMPERATURE STRESS RELAXATION AND TENSILE ADHESION TESTS

Apparatus

The apparatus suitable for performing these two tests can be any tensile testing machine capable of generating tensile stress of 2000 N, and with the ability to pull the sample at a minimum of two constant speeds - 1 mm/min and 10 mm/min. It also has to have the capacity to monitor the change in tensile stress. The apparatus has to be equipped with an environmental chamber able to accurately maintain the temperature of -30°C.

In our opinion, these requirements are sufficiently complicated to prohibit the construction of such an apparatus. We elected to use one of the machines available from a multitude of manufacturers (e.g. Instron, Tinius-Olsen, etc.)

Low Temperature Stress Relaxation Test

Mold Design

The mold assembly, shown in Figure 1, is drawn to scale. All the parts of the mold assembly are constructed using aluminum. The sections of the mold constructed with the tapered interior walls are done in a tiered fashion to reduce the change of slippage of the sample from the mold during the test. The part of the sample exposed to testing, after the removal of the split mold portion, is a cylinder of 25.4 mm height and 25.4 mm in diameter.

Material Preparation

Crack and joint sealant samples are usually collected from the manufacturers in the form sold to the customer. This is typically a 20 kg block and is sometimes susceptible to components (i.e. polymer, filler) migrating to the top or bottom. The manufacturer usually recommends that the entire block be heated and mixed before sampling. Since laboratories do not necessarily possess the facility to do that, the

alternative method is to sample the blocks in strips that go from the top to the bottom of the block. The strips are placed in individual containers totaling 500g per sample. The sample is then heated in the oven to the manufacturer's recommended safe heating temperature which is usually in the range of 180 to 195°C.

Mold Preparation

The mold consists of a top, bottom and two removable side pieces. The unit, as seen in Figure 1, shows the material in place and the cap on.

The removable sides are coated with silicone grease on the interior walls to act as a release agent. The unit is assembled and the "cap" is removed to allow the crack sealant to be added. When the crack sealant reaches the required temperature, it is immediately poured into the mold assembly up to the top. The sample container is returned to the oven and the mold assembly with the crack and joint sealant is allowed to cool. As it cools, the crack sealant contracts, leaving a depression or even a hole in the center. This is later filled with hot crack sealant and then the cap is threaded into place while the new material is still hot.

Sample Conditioning

The mold assembly with the crack sealant is placed into the freezer at -30°C for 16 hours (overnight). The middle pieces are then removed and the sample is clamped into the test apparatus located in an environmental chamber set at -30°C. The sample is conditioned for 60 minutes before testing.

Testing

The test apparatus (in our case, Materials Testing System manufactured by Instron) is computer controlled allowing for accurate stress rates, extensions and data collection.

The stress relaxation test is designed to take one hour to complete once the test is started. To accomplish this, the cross-head speed is set at 1 mm/min and travels a distance of 12.5 mm in 12.5 minutes to achieve a 50% extension of the sample. At the point of reading 12.5 mm the cross-head stops at the 50% extension point and the apparatus continues to measure the load generated by the sealant for the next 47.5 minutes. The load continues to drop as the material relaxes the stress generated by the extension.

Data Presentation

The data recovered from the apparatus (Instron) include two columns. While the stress is applied (extension period) the data are collected as "load vs. displacement". In the relaxation period, data are collected as "load vs. time". The Instron presented this data as load in poundforce (lbf), displacement in inches (in) and time in minutes (min). The data are converted to load in Newtons (N) and displacement in millimeters (mm). The load conversion is 1lbf = 4.4482N and the displacement conversion is 1in = 25.4mm. The displacement value is then converted to time by calculating the position as a function of time (i.e. 2.5 mm / 1.0 mm/min = 2.5 min). These new time values are then added to the relaxation time data (starting at 0) to give a 60 minute compilation of the application and relaxation of stress on the sample.

Calculation of Results

- a) The percent stress relaxation is calculated from the peak load during extension minus the relaxed load at 60 minutes divided by the peak load.

$$\% \text{ Stress Relaxation} = \frac{\text{Peak Load (extension)} - \text{Relaxed Load at 60 min}}{\text{Peak Load (extension)}}$$

- b) The alternative method to calculate stress relaxation is to determine the area under the stress relaxation part of the curve. The area under the relaxation part of the curve better describes the relaxation processes than the percentage of final relaxation.

To be able to compare stress relaxations of different crack sealants, the whole curve has to be normalized -- that is, every point of the curve has to be divided by the peak load.

Tensile Adhesion Test

Mold Design

The tensile adhesion mold assembly is constructed using a standard 19.12 mm (3/4") round headed bolt. A washer is used as the cap, fixed into position using standard 6.37 mm (1/4") hex nuts. The anchor immersed in the sample is also a standard 6.37 mm (1/4") hex nut. The anchor nut is necessary to prevent the bolt from pulling out of the sample before adhesion failure is achieved. The split mold is made out of aluminum and is 3.19 mm (1/8") thick with an inside diameter of 25.4 mm (1"). The mold is shown in Figure 2. The concrete bricks are the same as used in the ASTM D3407 Bond test.

Material Preparation

Crack sealant samples are usually collected from the manufacturers in the form sold to the customer. This is typically a 20 kg block and is sometimes susceptible to components (i.e. polymer, filler) migrating to the top or bottom. The manufacturer usually recommends that the entire block be heated and mixed before sampling. Since laboratories do not necessarily possess the facility to do that, the alternative method is to sample the blocks in strips that go from the top to the bottom of the block. The strips are placed in individual containers totaling 500 g per sample. The sample is then heated in the oven to the manufacturer's recommended safe heating temperature which is usually in the range of 180 to 195°C.

Mold Preparation

The mold consists of two removable sides of a cylinder that sit on top of a concrete block. The block conforms to ASTM D3407 (bond test). The removable sides are coated with silicone grease on the interior walls to act as a release agent and are held in place with a clamp. The anchor unit that goes into the center is preheated to 180°C. The assembly is shown in Figure 2.

The cylindrical mold is filled with crack sealant. When it reaches the required temperature the anchor is immediately inserted. The unit is cooled in one piece with no regard to contraction of the cooled material because of the ample amount used.

Sample Conditioning

The mold assembly with the crack sealant is placed in the freezer at -30°C for 16 hours (overnight). The middle pieces are then removed and the sample is clamped into the test apparatus, located in an environmental chamber at -30°C. The sample is conditioned for 60 minutes before testing.

Testing

The test apparatus (in our case, materials testing system manufactured by Instron) is computer controlled allowing for accurate stress rates, extensions and data collections.

The samples of crack sealant are pulled off the concrete brick at a rate of 10 mm/min. A plot of load versus displacement are recorded for each sample. Samples are tested in sets of four.

Data Presentation

The data recorded are:

- the maximum load required to remove the crack sealant from the concrete brick. This is typically obtained at the moment of release. If it is not, then it is noted that crack sealant peels off the brick rather than debonds;
- the work necessary for complete debonding. It is measured as the area below the curve of strength vs. extension;
- elongation of the sample at the point where the peak load is achieved;
- elongation of the sample at the point where the complete break occurs.

Similarly, as in the test for Low Temperature Stress Relaxation, the apparatus (Instron) presents the data in poundforce (lbf) for load and in inches (in) for displacement. Both are converted to the SI unit system.

5.4 DISCUSSION

Sixteen different commercially manufactured crack sealants were used to help design the stress relaxation and tensile adhesion tests. After the equipment design and testing conditions were determined, all sixteen crack sealants were tested by the new methods. Additionally, the following tests were performed on these materials: cone penetration at 25°C, resilience at 25°C, flow at 60°C, creep test at -30°C (stiffness modulus), viscosity at 190°C.

It is necessary to mention that the melting of the crack sealants for the sample preparation used in these tests has not been done according to the ASTM standard, but rather the crack sealants were heated to the required temperature in the preheated oven, after which they were poured for the different tests.

The cone penetration at 25°C, flow at 60°C and resilience at 25°C were performed according to the existing standards.

In addition to these tests, the creep of samples at -30°C was measured and the stiffness modulus at 500s calculated. The reason for using this test was to see whether there is a correlation between the results of the resilience test and this relatively simpler test. If there is a correlation between the results of the stress relaxation test and the stiffness modulus, this test could be used as a surrogate test .

The viscosity of crack sealants at 190°C was measured to more accurately determine the fluidity at the pouring temperature. As already discussed, there must be a balance between sufficiently low viscosity to produce good bond between the crack sealant and the crack wall by allowing the sealant to penetrate the crevices and pores of the crack wall and too low viscosity, which would cause the sealant flow out of the crack. Here, it is useful to mention that the viscosity of the more viscous materials increases rapidly with the decreasing temperature and can have a detrimental effect on the quality of the bond, especially when the sealing is performed at lower ambient temperatures.

The results of these tests are provided in Tables 1 and 2.

Stress Relaxation Test

The design and testing protocol for the stress relaxation test was already described. The test method was carried out on an Instron Model 4204 tensile testing machine equipped with an environmental chamber with liquid nitrogen cooling. The stress relaxation test generates a curve along with numerical data. The curves are depicted in Figures 3 through 18 and the numerical data is shown in Tables 1 and 2. When the crack sealant is stretched, the stress in material builds. The stress relaxation test, as it is defined in rheology, is performed so that deformation of the tested material is produced instantly, and there is no relaxation (internal flow of the material) during the loading period. This did not seem to be practical with existing equipment due to sample size and characteristics. The samples, therefore, were loaded as quickly as possible. It was decided to find the fastest speed to achieve the 50% extension of the sample without damaging samples during the extension period. Several cross-head speeds were evaluated and abandoned as too high, because high portion of samples was damaged.

Stress increased in the sealant during the initial 12.5 minutes of extension. After this time 50% extension of the sample is achieved.

Table 2 shows the percent of relaxation of the crack sealant after 47.5 minutes. The test was terminated after this time period because it was found that the majority of relaxation occurred in the first 47.5 minutes. Also, the relaxation which takes place in the first few minutes is, in our opinion, important as it discloses how quickly a crack sealant will reduce the stress between the sealant and the crack wall. The percentage of the stress relaxation (see Table 2), was measured at 1, 5, 10 and 47.5 minutes of the sealant relaxation period. The percentage of relaxation, after one minute for most materials was in 30 to 50% range.

The most important data produced in the stress relaxation test in this phase of the project were considered the peak loads reached during the stretching period. These peak loads for different crack sealants varied significantly from 63 N to 761 N. It was generally the softer materials (lower stiffness modulus at -30°C and cone penetration at 25°C) which produced the lower peak loads. The lower the peak load and the higher the percentage of stress relaxation of the sealant, the lesser should be the tension

exerted on the bond between the sealant and the crack wall and so the higher the chance of better performance of the sealant in the field.

Even though the test could not be designed in such a manner that there would not be relaxation of the material during the stretching period (12.5 minutes of stretching instead of an instantaneous deformation at a time as close to 0 as possible), the test as designed still allows rheological modelling of the crack sealant flow and so has a physical meaning. Because of the possibility of a quantitative interpretation, the crack sealants can be compared relatively to each other by several parameters and so marked as to the probability of their field performance.

The rheological interpretation of the test and comparison of the flow behaviour of different sealants will be a matter of a separate chapter.

Tensile Adhesion Test

The tensile adhesion test was designed to complement the test of stress relaxation. The test uses the same cross-sectional sample area for evaluation, however, the cross-head speed is 10 mm/min rather than 1 mm/min. The intention in using the higher cross-head speed for pulling the sample was to prevent the potential stress relaxation during the experiment.

The tensile adhesion test at -30°C produced load versus displacement curves of the pattern shown in Figures 19 through 34.

Figures 19 through 34 show the most representative curve from the parallel tests. Accompanying data sheets show the results of all measurements as well as mean data, calculated by the Instron software. As can be seen, some results show the label "excluded". These data were excluded on the basis of the physical condition of the sample as it was observed after the test was performed, i.e. cracks or any other physical damage of the sample, or the slippage of the grips. The exclusion of the results is based on the user's discretion. Excluded data are not used to calculate the "mean".

The load builds to a peak load, is maintained for a period of time (or length of stretching) and then a sudden release occurs when the bond between the crack sealant and the concrete brick breaks. The tensile adhesion test results revealed a bond failure at the sealant - brick interface in all cases except sample 2383. This material failed internally in the sealant, indicating that this material had a higher bond strength than cohesive strength.

Sample 2382 displayed different behaviour in the load versus displacement curve. The curve is characteristic of a sealant peeling slowly off the concrete brick after the peak load had been reached. The crack sealant 2382 showed an average of the maximum peak load at the sample elongation of 2.6 mm and an average maximum displacement at 14.2 mm. The material went into a ductile flow mode until the extension reached approximately 10 mm and then started to peel away from the concrete brick. The crack sealant 2382 is the same material as sealants 2449 and 2450 and the tensile adhesion curves of the other two show similar behaviour as the sealant 2382. However, samples 2449 and 2450 show a sharper bond break than sample 2382.

The tensile adhesion test at -30°C, using a cross-head speed of 10 mm/min, indicated which crack sealants displayed a tendency to flow under stress under fast loading. The occurrence of the ductile flow indicates that the sealant will have an increased resistance to debonding under shock loading at low

temperatures. To interpret the results of the tensile adhesion test properly, the whole curve of the load versus displacement should be taken into consideration as well as the extension at break. The extension at peak load can vary among individual tests on the same crack sealant but the overall profile of the curve is similar, indicating the behaviour is much the same.

Originally, it was considered that concrete bricks would be replaced by bricks from asphalt mixes. Some initial testing was undertaken, however, it became clear that development of a standard asphalt brick is beyond the scope of this project.

5.5 CONCLUSIONS

Within the scope of the second phase of this project, two tests for evaluation of crack sealants - the stress relaxation test and the tensile adhesion test were developed. The necessary equipment was constructed and the testing protocol established.

Sixteen different crack sealants were evaluated by the two newly developed tests as well as by the tests of viscosity at 190°C, stiffness modulus at -30°C and by three tests present in existing specifications (ASTM D3407 and others) - cone penetration at 25°C, resilience at 25°C and flow test at 60°C.

The evaluation of different crack sealant demonstrates that:

- the newly developed tests show a promise in evaluation and differentiation between the different crack sealants and in quantification of their properties;
- there is a considerable difference in behaviour of crack sealants in their responds to stresses at low service temperatures.

From the testing of the crack sealants the following **preliminary** guidelines were established as to their properties at the installation temperature and the different service temperatures. The guidelines are summarized in the following table.

Preliminary Guidelines for Further Test Development

1. Viscosity at 190C	<ul style="list-style-type: none">• < 3 Pa·s
2. Cone Penetration at 25C	<ul style="list-style-type: none">• >100 dmm• <160 dmm
3. Flow at 60C	<ul style="list-style-type: none">• < 8 mm
4. Stress Relaxation at -30C	<ul style="list-style-type: none">• High level of relaxation• Low peak load
5. Tensile Adhesion at -30C	<ul style="list-style-type: none">• Low peak load• Long extension with minimum load growth• Long displacement before break or debonding occurs

In the third phase of the project a more thorough rheological interpretation of the two new tests was performed and thus some more exact parameters to compare the different crack sealant were developed.

**Laboratory Evaluation
of Crack Sealants
Used in the Field Projects**

6.1 INTRODUCTION

Phase II of the project "Laboratory Testing of Crack Sealing Materials for Flexible Pavements" was concluded in January 1995. The third phase dealt with testing of 14 different crack sealants which were used and monitored in field projects in Alberta, Ontario and Quebec by newly developed tests of stress relaxation and tensile adhesion. The results were compared with the performance of crack sealants in the field. It was also decided that rheological analysis of different flow patterns would be performed.

In October and November 1995, the 14 selected crack sealants used in field projects arrived. Eight materials were provided by the Province of Alberta, five materials by the Province of Ontario and one material by the Province of Quebec. These materials were tested by the stress relaxation test and by the tensile adhesion test. Additionally, viscosity at 190°C was measured as well as the stiffness modulus at -30°C. Rheological analysis of the results was also done.

Results from laboratory testing were compared with reported field performance of the materials.

The stress relaxation test for the crack sealants was analyzed for its rheological meaning. As a part of this analysis the stress relaxation curves were normalized so that they could be compared. This allowed also for better evaluation of the relaxation period. Instead of determining the percentage of the recovery at the end of the test, the area under the recovery curve was measured. This area reflexes also the speed of the relaxation and not only its final value. The rheological interpretation of the stress relaxation test is attached in Appendix A.

6.2 TESTING OF CRACK SEALANTS USED IN FIELD PROJECTS

Fourteen different crack sealants were obtained from three provinces: eight from Alberta, six from Ontario and one from Quebec. A list of all materials is given in Table 3. The table also summarizes in what form the crack sealants were obtained. (Sampling remarks). These materials were used in field trials or in normal jobs and their performance observed in most cases over the one year period. In two cases, Ontario reported installations three years old and, in one case, installations two years old.

The fourteen materials provided by the three provinces were tested by the stress relaxation and tensile adhesion tests according to the testing protocol described in Phase II of this report. Results of tests are summarized in Tables 5, 6, and 7 and Figures 36 through 66.

The difference in testing procedure relative to that described in Phase II of this report was in melting the sealants for the sample preparation. In this set of tests, the ASTM standard was strictly adhered to. A melting apparatus capable of melting three different crack sealants at the same time was designed and constructed. The design of the apparatus is sketched in Figure 35.

Viscosity of crack sealants at 190°C was measured by rotational viscometer Contraves and the results are provided in Table 5. Each test was performed only once.

Stiffness modulus at -30°C of crack sealants was measured on Enraf Nonius Sliding Plate Rheometer and the results are summarized in Table 6. Stiffness modulus was also tested for each sample of crack sealant only once. An attempt was also made to measure the stiffness modulus on the Bending Beam

Rheometer, developed within the Strategic Highway Research Program (SHRP). However, because of greater flexibility of crack sealants at this temperature, relative to paving asphalts, deformation of the beams immediately became very large. The BBR is not suitable to measure stiffness of crack sealants.

6.3 DISCUSSION

In Phase II of this project, two tests for evaluation of crack sealants were developed - the stress relaxation test and the tensile adhesion test. The description of the tests and the Testing Protocol are part of this report. Within Phase II of the project, 16 crack sealants were tested by these tests, as well as by some other tests, those being either tests required by different specifications (resilience, flow, cone penetration), or tests decided upon in our laboratory (stiffness modulus at -30°C, viscosity at 190°C). The results and preliminary recommendations are described more closely in the "Progress Report on Tasks 3 & 4 - Laboratory Testing Procedures & Instrument Design", authored by R.D. Watson and D. Sieben. Field performances of the 16 crack sealants tested within that sequence of the project were not known and the comparison could not be done.

In Phase III of this project 14 crack sealants used in the field were provided. Eight came from Alberta, five from Ontario and one from Quebec. Sampling remarks are provided in Table 3.

These fourteen crack sealants were used either for normal jobs, or as field trials and most were inspected for their performance after one year in service, three possibly after two or three years in service.

These fourteen crack sealants were subjected to testing by the stress relaxation and tensile adhesion tests. Also, their viscosity at 190°C and their stiffness modulus at -30°C and 500s loading time were measured.

The results of the stress relaxation test are given in Table 4 and in Figures 36 through 49. Table 4 summarizes the results from the stress relaxation test. This test was performed for each material in duplicate. When results were apart or there was suspicion of any irregularity, the third sample was tested. In Figure 36 through 49 one curve for each material is shown for demonstration.

The results from the tensile adhesion test are given in Figures 50 through 63.

The tensile adhesion test was performed generally on four samples. After the test was performed, the samples were observed for their physical condition. If any cracks or other physical damage on the samples was found, or slippage of the grips occurred, the data obtained from this sample were excluded. The exclusion of the results was based on the user's discretion. Excluded data was not used to calculate the "mean" by the apparatus software. Results are shown in Figures 50 through 63 accompanied by the raw results as produced by the apparatus software. In most cases, the plotted curve represents the "mean". However, in the case of crack sealants with the maximum displacement lower than 1 mm, all curves are shown (Figures 56, 57, 59, 62). In these cases, the mean does not show a good description of the curves. (In the case of sample 9, Fig. 58, the software was able to calculate and plot the mean).

Generally, the repeatability of the stress relaxation test was very good through the testing. The repeatability of the tensile adhesion test was lower and therefore four samples (instead of two as in the stress relaxation test) were used. The lower repeatability of the tensile adhesion test may be explained by

the fact that the sealant was pulled from the concrete surface instead of just being stretched as in the case of the stress relaxation test. This introduces some level of subjectivity. In the case of the stress relaxation test, strictly flow process is taking place. In the case of tensile adhesion, separation of two surfaces combined with the flow is observed. However, the repeatability is still reasonable and the test is fast and simple and can be easily repeated for sufficient amounts of sample to obtain credible results. The advantage relative to the present bond test is also that the result is numerical as opposed to the pass-fail result in the present bond test.

When attempting to predict the performance of crack sealants in service, one should always bear in mind that beside the properties of the crack sealant, there are other factors which cannot be predicted. Some of these factors are the quality of installation, type of the crack, type of paving mix, weather conditions, water table level, intensity of traffic etc. These factors were discussed in previous parts of the report. Therefore, we can only attempt to predict the potential performance of the crack sealant which depends on its characteristics if all the external influences are the same. We have a better chance of predicting sealant failure, if the crack sealant has some negative characteristics, rather than success which also depends on external factors. Also, there is the possibility that a well installed, lower quality material may perform better than a poorly installed, higher quality material under a multitude of adverse conditions.

The different parameters produced by these tests were assessed for their importance in predicting the potential performance of crack sealants. Four parameters were selected -- two from the stress relaxation test and two from the tensile adhesion test.

The two parameters from the stress relaxation test are the peak load and the level of relaxation. It was thought that a lower peak load would decrease material stiffness and therefore give the material a better chance to stretch at low temperatures without any damage. The peak load is automatically measured by the Instron machine and given in Newtons.

The area under the stress relaxation part of the curve would indicate the ability of the material to relax the stresses after stretching. The less residual stress, the lower the tendency of the crack sealant to debond or crack. This area under the relaxation part of the curve better describes the relaxation processes than the percentage of final relaxation. To be able to compare the areas under the stress relaxation part of the curve, the whole curve has to be normalized; that is, every point of the curve has to be divided by the maximum peak load. In our case, we obtained the normalized curves when rheologically analyzing the relaxation curves. However, this form of curve can be obtained simply without need of any mathematical apparatus used in the rheological analysis. The area under the relaxation part of the curves for all 30 tested crack sealants is given in Tables 1 through 3 in the Appendix. In Table 3 of the Appendix the crack sealants are put into order according to increasing area under the relaxation part of the curve. Both parameters used from the relaxation test should have the least possible value.

If the two parameters from stress relaxation -- the peak load and the percentage of relaxed stress -- are weighted it appears that the more important factor is the peak load (it actually includes some stress relaxation which occurred during the stretching period). However, one can assume that from two crack sealants with the same peak load, the material with higher level of stress relaxation stands a better chance of not debonding.

Two parameters selected from the tensile adhesion test were the maximum extension of the crack sealant at the moment of completely breaking and the work necessary for debonding of the crack sealant from the wall. (There was no case when the failure occurred within the crack sealant.) It was assumed that the extension of the crack sealant before debonding indicated its willingness to flow at low temperatures, therefore giving the material a better chance to adjust its shape when the crack widened in cold periods.

The work necessary for complete debonding should indicate the capability of the material to adhere to the wall under stress and to resist debonding.

Both parameters from the tensile adhesion tests should have values as large as possible.

Both these parameters are obtained directly from the reading provided by the Instron testing machine. Maximum extension is given in millimeters and the machine itself subtracts the original period where the extension does not occur (except in two cases during the whole testing). The work necessary for debonding is the area under the curve and is described as toughness and given in Joules.

During testing of the 14 materials, it was found that several samples broke during the stress relaxation test. By analyzing results from the stress relaxation tests of the first 16 materials, it was found that some of these also had likely broken. The materials which probably broke were identified by rheological analysis and are discussed in the Appendix.

Comparison of the Field Performance of Crack Sealants With Laboratory Tests Results

After obtaining reports on the field performance of crack sealants, data was correlated with selected parameters from the stress relaxation and tensile adhesion tests. The report from Alberta Transportation provided descriptions and performance ranking of crack sealants. The best performing crack sealant was ranked as 0, the worst performing crack sealant as 5. Materials which ranked 4 and 5 were considered as failed. The report from Ontario Ministry of Transportation did not rank by numbers, but rather by description. Failure was considered when more than 10% of the crack sealant debonded. For twelve out of fourteen crack sealants evaluated within this phase, the field performance report was obtained.

The performance report on the individual crack sealants was compared with the four parameters described above. The comparison can be seen in Table 7 and on Figures 64 through 66.

Table 7, compiled from field reports, shows that from those crack sealants, for which field performance evaluations are available, four can be considered failures in the field: crack sealants 7, 8, 11, and 13.

Peak loads from stress relaxation tests of the 14 crack sealants used in field trials were spread between 75.2N and 1243.0N. When peak loads of the four materials that failed in the field are compared with the peak load of other satisfactorily performing crack sealants, we can see that those crack sealants which failed in the field had peak loads above 500N and broke during the stress relaxation test.

From the group of 14 field tested crack sealants, five broke during the stress relaxation test. As already mentioned, all four crack sealants which failed in service belong to this group. The fifth material, No.3, also broke; however, this material is reported to have satisfactory performance in the field.

It can be discussed that if loading were applied slower than 1 mm/min, the materials would not break and if loading were applied faster, more materials would break. However, at this particular loading speed all materials which failed in the field had peak loads higher than 500N and subsequently broke.

Crack sealants which failed in the field and which also broke in the stress relaxation test prevented comparisons between the relaxation capability of these materials and crack sealants which succeeded in the field. The relaxation part of the curve for crack sealants which broke during the extension period is therefore meaningless.

The maximum extension of crack sealants at the moment of complete debonding for the 14 materials, tested in the laboratory and evaluated in the field, had ranges from 0.31 mm up to 68.99 mm (Table 7, Figure 65). When the values of this parameter are compared with the field performance report, two groups of crack sealants emerge. One group, with a maximum extension lower than 1 mm, included only those four crack sealants that were reported as failures in field trials. All other crack sealants, which have a maximum extension 5 mm and up, belong to the group with satisfactory field performances. There was no crack sealant with a maximum extension between 1 mm and 5 mm from those evaluated in the field, so interpretation of this extension could not be done.

A similar pattern emerges when the work necessary for debonding is compared with the field reports (Table 7, Figure 66). The work necessary to debond the crack sealant for the group of four materials reported as failures in the field is less than 1J. All other crack sealants are above 1J, most above 5J. The difference in this value for the satisfactory performing materials and failures according to this parameter is not as sharp, as in the case of the maximum extension.

The collection of 12 crack sealants, for which the reports on field performance are available, is too small to quantify the parameters further especially when all other factors which impact on field performance cannot be taken into account. It is therefore difficult to say whether there is further difference in the performance of two crack sealants which, for example, have maximum extensions of 7mm or 20mm at debonding. It is possible to say that there is a critical value of the selected parameter. If the crack sealant does not meet this critical parameter, the likelihood of the field failure is considerably increased. Values can be considered as follows:

1. All four crack sealants which failed in the field had maximum peak loads above 500N. They represented two-thirds of the crack sealants which had maximum peak loads above 500N.
2. All four crack sealants, which broke during the extension period of the stress relaxation test, failed in the field. They represented 80% of crack sealants which broke during the test.
3. All four crack sealants, which had the maximum extension at debonding during the tensile adhesion test lower than 1 mm, failed in the field. All other crack sealants had the maximum extension of 5 mm and more.
4. All four crack sealants, which had the total work needed for debonding lower than 1J failed in the field. All other crack sealants had the total work necessary for debonding close, or above 2J.

Therefore, it can be suggested that if crack sealant belongs to one of these categories (breaking during the extension in stress relaxation test, maximum extension at debonding during the tensile adhesion test less than 1 ~ 5 mm and work necessary for debonding less than 1J), the likelihood of its

failure in the field increases considerably. If the crack sealant belongs to more than one of these categories, its use for crack sealing is not advisable.

Beside the stress relaxation and tensile adhesion test, the crack sealants were also evaluated for their viscosity at 190°C. In our opinion, the safe heating temperature described in the specifications should refer more to the heat stability of the polymers used in crack sealant formulation, than to the temperature which should be used during the installation. Viscosity of crack sealants, especially those with high viscosities during installation, increases rapidly with a decrease in temperature, which may cause poor filling of the crack (especially capillaries and crevices in the crack walls) with sealant, thus creating a weaker bond. The introduction of a maximum viscosity at installation temperatures into specification may therefore be considered.

Table 5 lists the viscosities of crack sealants from the field. One can see that satisfactory performance was obtained with crack sealant of very different viscosity at 190°C. Also, the viscosities of materials which failed are among the highest and lowest in the group. However, the pouring of crack sealants with viscosity at 190°C higher than 3000 mPa·s was difficult. One possibility would be to define the difference between a safe heating temperature as provided by the manufacturer and a temperature at which the crack sealant's viscosity is 3000 mPa·s.

The stiffness modulus of crack sealants at -30°C was also measured by a sliding plate rheometer. The idea was to discover if stiffness modulus can be used as a surrogate test for the two new tests. The data on stiffness modulus are summarized in Table 6. It should be noted that in some cases the stiffness modulus at -30°C could not be measured and instead, the value of stiffness modulus at -20°C is given. The data show a definite pattern toward higher stiffness of materials which failed in the field. The difficulty of measuring the stiffness modulus at -30°C of the harder materials on a sliding plate rheometer and the value differences, which at this temperature can be considered relatively small, will probably disqualify this test. Using the Bending Beam Rheometer resulted in failure, because it was impossible to measure the stiffness modulus of more flexible samples.

**Summary, Conclusions
& Recommendations**

7.0 SUMMARY, CONCLUSIONS AND RECOMMENDATIONS

The study to develop a new, simple laboratory testing method or methods capable of predicting the field performance of crack sealants was carried out in three phases. In the first phase, the literature research was performed to assess the present situation in the crack sealant testing and specifications. In the second phase, two methods for the evaluation of the low temperature behaviour of crack sealants were designed, the necessary equipment constructed and a slate of crack sealants tested. In the third phase, a group of crack sealants used in field applications in three provinces were selected and tested by the newly developed methods. The service performance of the crack sealants was compared with laboratory tests results. Analysis and interpretation of the rheological behaviour of crack sealants in the newly developed test was done. A summary and major conclusions are as follows:

A. Assessment of tests in existing crack sealant specifications

1. The present standards for crack sealants are not sufficiently clear and restrictive about the fluidity of crack sealants at installation temperatures. It is proposed to measure viscosity at 190°C by rotational viscometer. The maximum allowable viscosity at this temperature should be established.
2. When specification contains a restriction on the softness of crack sealant at highest service temperatures (maximum flow at 60°C) and requires set of properties at low service temperatures, the cone penetration at 25°C is to some degree redundant. Limiting the cone penetration to maximum of 90 dmm may eliminate good crack sealants. Establishment of upper and lower limits in a relatively wide range should be considered.
3. The resilience test is artificial. Crudely measured rebound does not appear to have any direct relationship to the field performance of the crack sealant.
4. The present bond test appears to be unreliable, with a low repeatability and no relationship with the field performance. Its replacement with more appropriate test should be considered.

B. Development of new test methods for crack sealants

1. Two test methods were designed to evaluate the low temperature characteristics of crack sealants. One method measures the stress relaxation characteristics of the crack sealant - the stress relaxation test; the second method measures the bonding characteristics of the crack sealant - the tensile adhesion test.
2. Necessary equipment to perform the two new tests was designed and constructed.
3. Preparation and conditioning of the samples was determined; optimum test parameters (temperature, cross-head speed, relaxation time etc.) were identified using a slate of commercially manufactured crack sealants.
4. Testing protocol was completed.
5. Sixteen commercially manufactured crack sealants were evaluated for their cone penetration at 25°C, resilience at 25°C, flow at 60°C, stiffness modulus at -30°C and viscosity at 190°C. Stress relaxation and tensile adhesion tests were performed.

6. Preliminary guidelines for the characteristics of crack sealants were developed.

C. Relation between the laboratory test results and the field performance of crack sealants

1. Fourteen crack sealants used in three provinces (Alberta, Ontario, Quebec) in the field were evaluated in the laboratory by stress relaxation test and tensile adhesion test. Viscosity at 190°C and stiffness modulus at -30°C were also determined.
2. From 14 selected crack sealants, reports on the field performance of 12 are available. From these four failed and eight performed well or satisfactorily.
3. Two parameters from the stress relaxation test: a) peak load, b) area under the relaxation part of the curve and two parameters from the tensile adhesion test: a) maximum elongation at debonding, b) work necessary for debonding were selected for comparison with the field performance data.
4. Stress Relaxation Test: There appears a limiting value of the peak load, above which the crack sealants break during the test. This peak load appears to be between 500N and 600N. Breakage of the crack sealant during the extension period correlates well with its field performance. All four crack sealants which cracked during the test failed in the field. Five crack sealants out of 14 cracked during the test.
5. Tensile Adhesion Test: Maximum extension of crack sealants at debonding correlates well with the field performance. All crack sealants which failed in the field have the maximum extension of less than 1 mm. All others have a maximum extension of at least 5 mm. The work necessary for debonding of the crack sealant correlates well with the field performance. All crack sealants which failed in the field have the necessary debonding work less than 1J. All others have the necessary debonding work close to 2J and more.
6. Stiffness modulus at -30°C: The stiffness modulus at -30°C shows a higher tendency for those crack sealants that failed in the field. However, the differences are sometimes relatively small and the measurement of all crack sealants at one temperature on one apparatus is difficult. The test therefore is not a very good tool to predict the crack sealants' performance.

It can be concluded, that the reported field performance of the twelve crack sealants correlates well with the following parameters of the two designed tests.

**Parameters from the Stress Relaxation and Tensile Adhesion Tests
Correlating with Field Performance**

TEST	PARAMETER	PASS	FAIL
Stress Relaxation	Maximum Peak Load	less than 500N	more than 500N
	Breakage of the crack sealant during extension period	does not break	breaks
Tensile Adhesion	maximum extension at debonding	above 5 mm	below 1 mm
	work necessary for debonding	above 2J	below 1J

D. Rheological analysis of the stress relaxation test

1. The rheological conditions of the relaxation test in both extension and relaxation part were defined.
2. All tested crack sealants (from both the second and third phase of the project) were evaluated and their behaviour described by the stretch exponential function.
3. The rheological analysis of the behaviour of crack sealant during the stress relaxation tests points to different types of flow among different crack sealant. Part of the materials behaved as linear viscoelastic liquids, others manifested at the same conditions non-linear viscoelastic behaviour, which probably is identical with the reported "ductile" flow.

E. Recommendations

Two methods for the evaluation of the low temperature field performance of crack sealants have been developed. In the group of twelve commercial crack sealants with known field performance, they were able to select the crack sealants which failed in the field.

The following course of action is recommended:

1. The two developed tests should be used to evaluate larger amounts of crack sealants with known field performance and in different laboratories. This will allow to assess the validity of the new testing methods on a broader basis and to better quantify the values of selected parameters.

2. If the broader based testing confirms that the newly developed tests are able to better predict the field performance than tests in existing specifications, particularly the bond test and the flexibility test, the change of the CGSB-37.50-M89 specification should be considered as follows:

- replacement of the bond test and flexibility test by the Stress Relaxation Test and Tensile Adhesion Test;
- raising the limit for the cone penetration test at 25°C;
- introduction of the maximum viscosity (by rotational viscometer) at 190°C.

Tables & Figures

TABLE 1. Test Results of Various Crack Sealants Using Conventional and Developed Test Methods

Sample #	Cone Penetration at 25C [dmm]	Resilience at 25C [%]	Flow at 60C [mm]	Stiffness Modulus at -30C [N/m ²]	Viscosity at 190C [mPa·s]	Tensile Adhesion at -30C			Stress Relax. At -30C		
						Peak Load [N]	Displcmt. Peak Load [mm]	Displcmt. At Break [mm]	Relaxation (50% Extn.) [%]	Peak Load [N]	Relaxed Load [N]
2379	74	62	1	5.47E+05	6171	412	9.9	15.1	62	98	37
2380	74	87	< 1	1.85E+06	2203	523	9.5	11.4	53	243	115
2381	156	58	5	7.73E+06	428	622	27.7	30.1	76	176	42
2382	64	69	2	1.08E+07	4360	590	2.6	14.2	81	250	47
2383	72	67	1	1.00E+08	960	748	0.4	0.4	95	648	53
2384	108	78	< 1	1.01E+06	5132	327	18.0	30.3	92	63	5
2385	87	64	2	5.14E+06	5619	1027	1.5	1.4	82	474	85
2386	74	68	2	2.00E+07	5354	1023	0.4	0.4	78	761	164
2387	93	54	2	> 1e8	1087	818	0.4	0.4	79	739	157
2388	115	40	1	5.00E+07	630	591	4.0	34.3	80	137	28
2389	69	62	1	2.24E+06	2366	731	22.0	24.4	67	155	51
2390	64	59	1	8.38E+06	6411	1145	1.6	1.8	89	463	52
2448	73	66	< 1	5.00E+07	N/A	323	8.4	27.6	90	340	34
2449	81	81	< 1	3.60E+06	3716	346	6.8	15.6	68	150	48
2450	68	72	1	2.40E+07	3990	365	5.7	13.5	59	187	76
2451	124	78	2	1.31E+06	934	329	10.3	27.1	70	141	43

TABLE 2. Results of Stress Relaxation Test at Various Time Intervals During Relaxation Period

Sample #	Stress Relaxation at -30°C											
	1 [min.]			5 [min.]			10 [min.]			47.5 [min.]		
	Relaxation (50% Extn) [%]	Peak Load [N]	Relaxed Load [N]	Relaxation (50% Extn) [%]	Peak Load [N]	Relaxed Load [N]	Relaxation (50% Extn) [%]	Peak Load [N]	Relaxed Load [N]	Relaxation (50% Extn) [%]	Peak Load [N]	Relaxed Load [N]
2379	23	98	75.5	40	98	59	49	98	50	62	98	37
2380	19	243	196	33	243	162	40	243	145.5	53	243	115
2381	30	176	123	52	176	84	62	176	67	76	176	42
2382	32	250	171	50	250	126	58	250	105	81	250	47
2383	46	648	352	69	648	200	79	648	138	92	648	53
2384	37	63	39.7	62	63	24	74	63	16.6	92	63	5
2385	35	474	308	59	474	195	68	474	150	82	474	85
2386	37	761	481	57	761	325	66	761	256	78	761	164
2387	36	739	475	57	739	315	66	739	250	79	739	157
2388	37	137	87	58	137	58	67	137	45	80	137	28
2389	28	155	111	45	155	85	54	155	71	67	155	51
2390	38	463	286	64	463	169	74	463	122	89	463	52
2448	60	340	136	74	340	89	80	340	69	90	340	34
2449	29	150	107	46	150	81	55	150	67	68	150	48
2450	24	187	143	39	187	115	47	187	100	59	187	76
2451	23	141	109	41	141	83	52	141	68	70	141	43

TABLE 3. Crack Sealants Supplied From Field Projects

CRACK SEALANT	SAMPLING REMARKS
1	<ul style="list-style-type: none"> • poured from block
2	<ul style="list-style-type: none"> • cut from block • 94-05-12
3	<ul style="list-style-type: none"> • cut from block • 94-05-11
4	<ul style="list-style-type: none"> • cut from block • 95-10-17
5	<ul style="list-style-type: none"> • cut from block • 95-02-10
6	
7	<ul style="list-style-type: none"> • cut from block • 94-05-11
8	<ul style="list-style-type: none"> • cut from block • 94-10-25 • thin layer of plastic which covers slab is mixed in with sample
9	<ul style="list-style-type: none"> • poured from block
10	<ul style="list-style-type: none"> • poured from block
11	<ul style="list-style-type: none"> • 2 samples cut from block
12	<ul style="list-style-type: none"> • poured block
13	
14	<ul style="list-style-type: none"> • cut from block • 95-10-12

TABLE 4. Stress Relaxation Test of Crack Sealants from Field Trials - Summary of Results

Crack Sealant	Run	Peak Load [N]	End Load [N]	Relaxation [%]	Run Conditions	
1	1	501.5	125.5	75.0		
	2	511.2	133.4	73.9		
2	1	209.1	68.4	67.3		
	2	192.3	65.1	66.2		
	3	216.8	65.5	69.8		
3	1	1221.5			Broke	
	2	1069.1				Broke
4	1	269.8	64.7	76.0		
	2	305.4	82.1	73.1		
5	1	76.7	26.2	65.9		
	2	73.6	26.2	64.4		
6	1	334.6	80.5	75.9		
	2	371.8	90.0	76.1		
	3	340.7	93.7	72.5		
7	1				Broke	
	2	610.2				Broke
8	1				Broke	
	2	1243.0				Broke
9	1	1175.8			Broke	
	2	1304.0				Broke
10	1	311.5	79.3	75.5		
	2	297.7	76.6	74.3		
11	1	684.6	138.9	79.7		
	2	566.7	136.2	76.0		
	3	782.5	164.6	79.0		
12	1	132.6	51.5	61.1		
	2	132.1	52.1	60.6		
13	1	662.3			Broke	
	2	553.0				Broke
	3	558.4				Broke
14	1	130.3	40.5	68.9		
	2	123.4	38.7	68.6		

TABLE 5. Viscosity at 190°C of Crack Sealants from Field Trials

Crack Sealant	Viscosity at 190°C [mPa·s]
1	4116
2	2817
3	798
4	1088
5	4289
6	812
7	8383
8	421
9	3140
10	1975
11	1397
12	3755
13	2833
14	N/A

**TABLE 6. Stiffness Modulus at -30°C of Crack Sealants from Field Trials
(500s Loading Time)**

Crack Sealant	Testing Temperature [°C]	Stiffness Modulus [N]
1	-30°C	2.27×10^7
2	-30°C	1.18×10^6
3	-30°C	3.40×10^7
4	-30°C	1.36×10^6
5	-30°C	5.90×10^5
6	-30°C	1.84×10^7
7	-30°C -20°C	3.67×10^7 7.09×10^6
8	-20°C	6.40×10^7
9	-20°C	3.69×10^6
10	-30°C	4.64×10^6
11	-20°C	4.70×10^6
12	-30°C	4.16×10^5
13	-30°C	3.96×10^7
14		

TABLE 7. Comparison of Selected Parameters from Stress Relaxation and Tensile Adhesion Tests with Field Performance of Crack Sealants

Crack Sealant	Ranking	Stress Relaxation Test		Tensile Adhesion	
		Peak Load [N]	Relaxation [area]	Extension [mm]	Work [J]
1	satisfactory	506.5	959.6	11.02	9.91
2	satisfactory	206.1	1169.7	6.90	2.42
3	satisfactory	1145.0	broke	5.01	1.82
4	satisfactory	287.6	988.2	12.35	6.80
5	satisfactory	75.2	1240.2	20.12	5.43
6	satisfactory	349.0	958.8	10.97	5.28
7	failed	610.2	broke	0.31	0.16
8	failed	1243.0	broke	0.44	0.13
9	no evaluation	1239.9	broke	0.31	0.17
10	satisfactory	304.6	977.8	17.40	17.07
11	failed	677.9	915.0	0.58	0.40
12	satisfactory	132.3	1358.7	22.14	6.22
13	failed	555.7	broke	0.90	0.51
14	no evaluation	126.8	1181.9	68.99	14.35

FIGURE 1
Sample Mold for Stress Relaxation Test

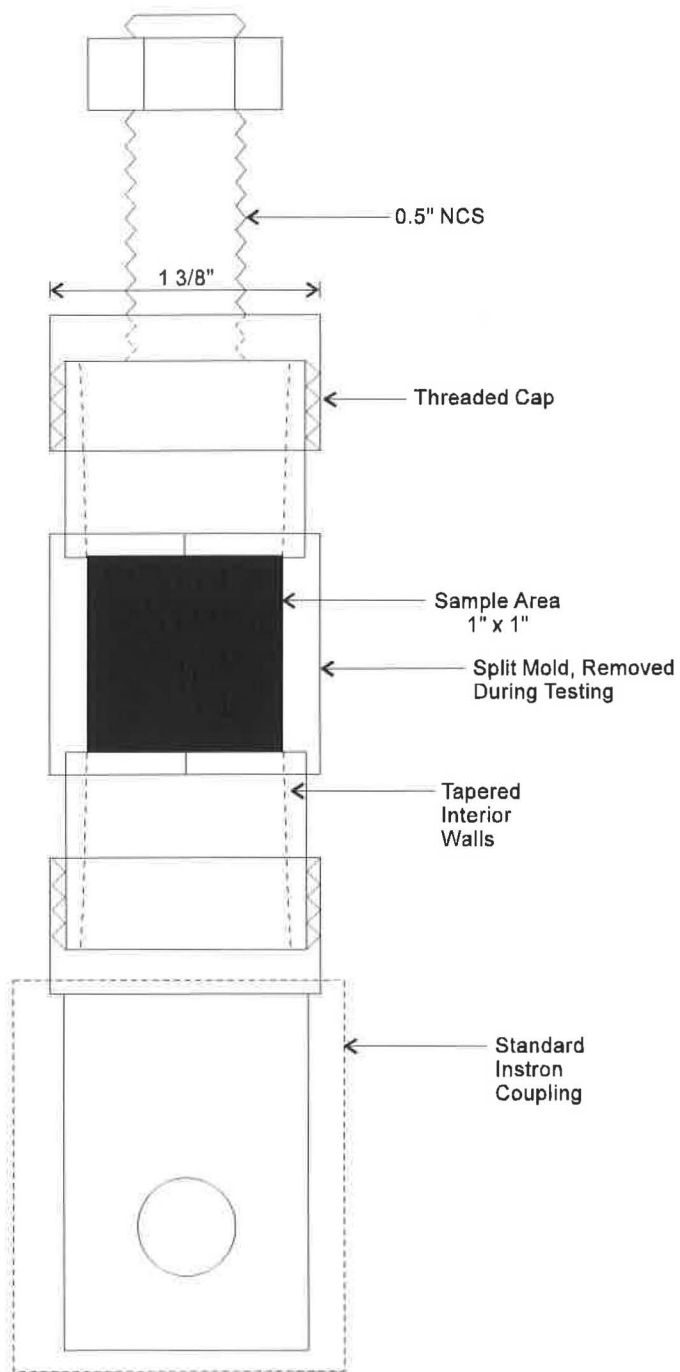


FIGURE 2.
Sample Mold for Tensile Adhesion Test

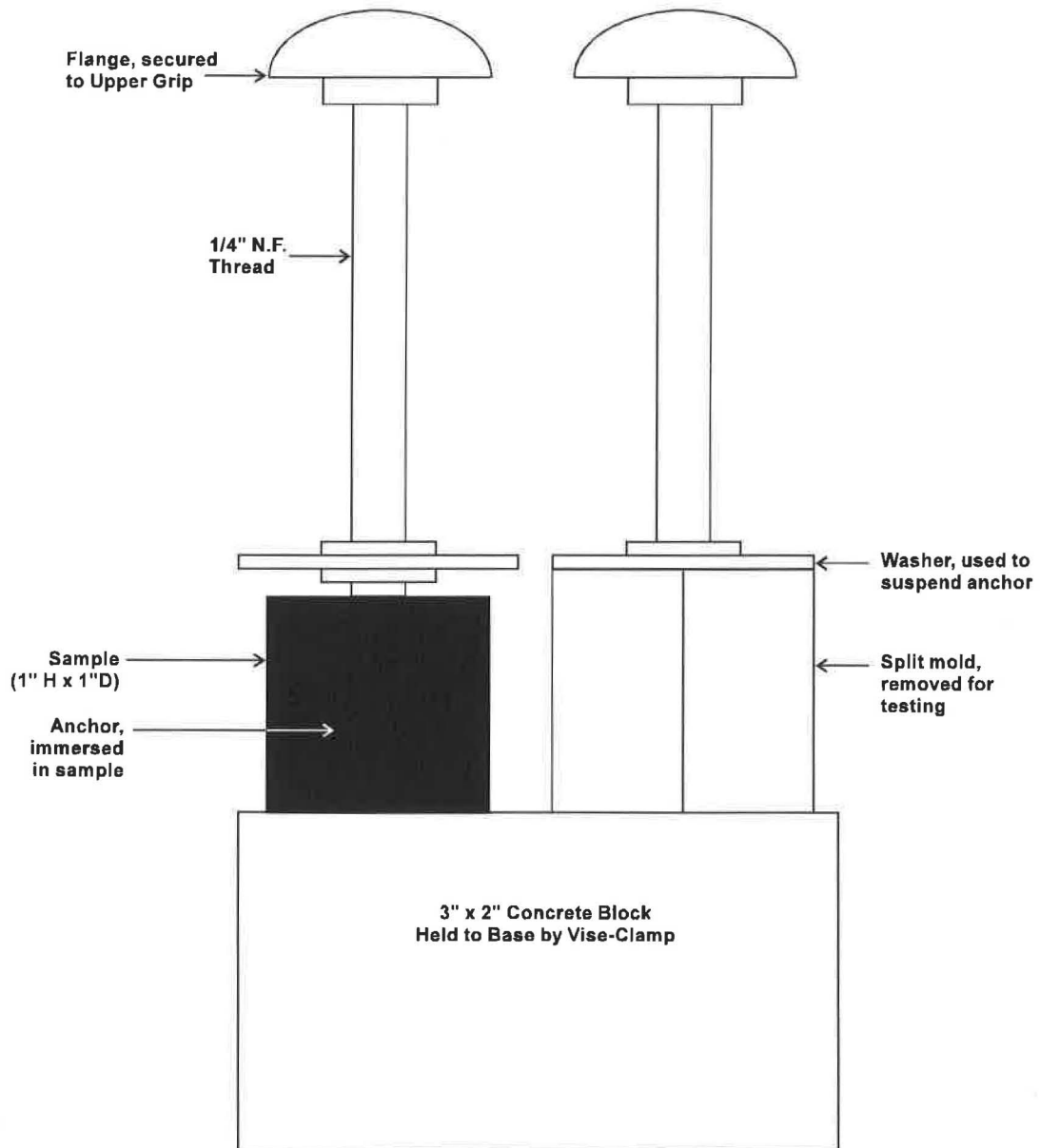


FIGURE 3. Stress Relaxation at -30°C, Sample 2379

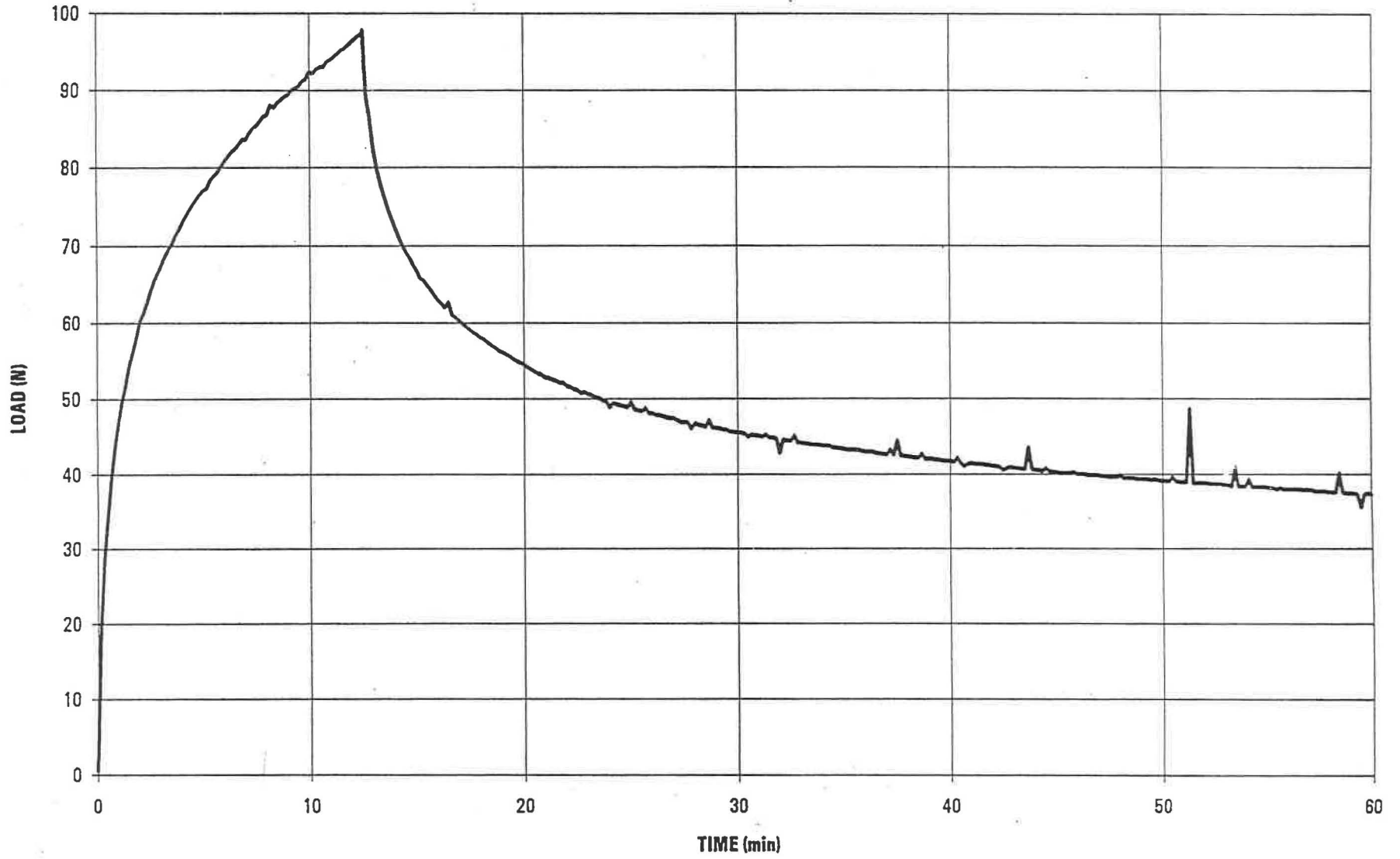


FIGURE 4. Stress Relaxation at -30°C, Sample 2380

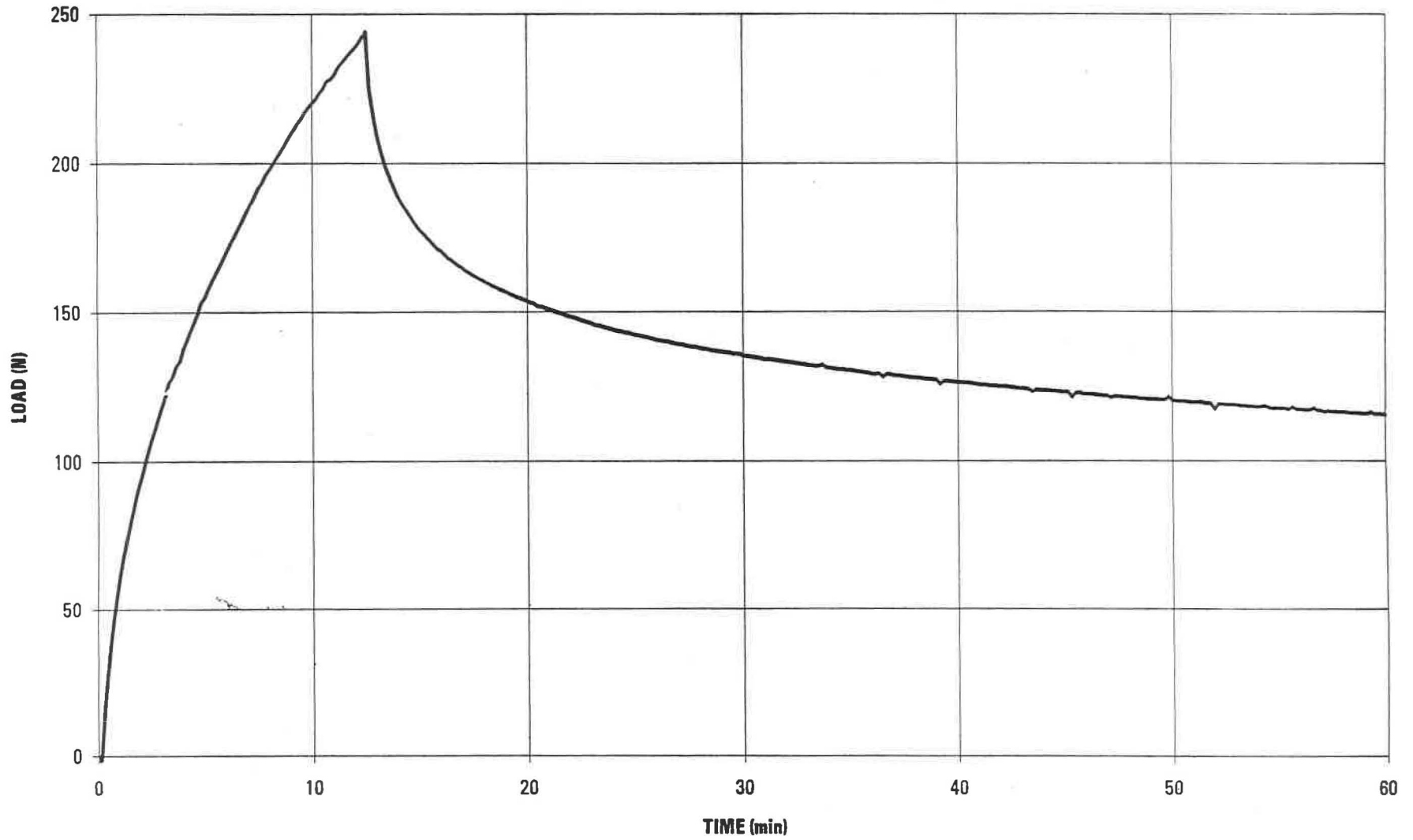


FIGURE 5. Stress Relaxation at -30°C, Sample 2381

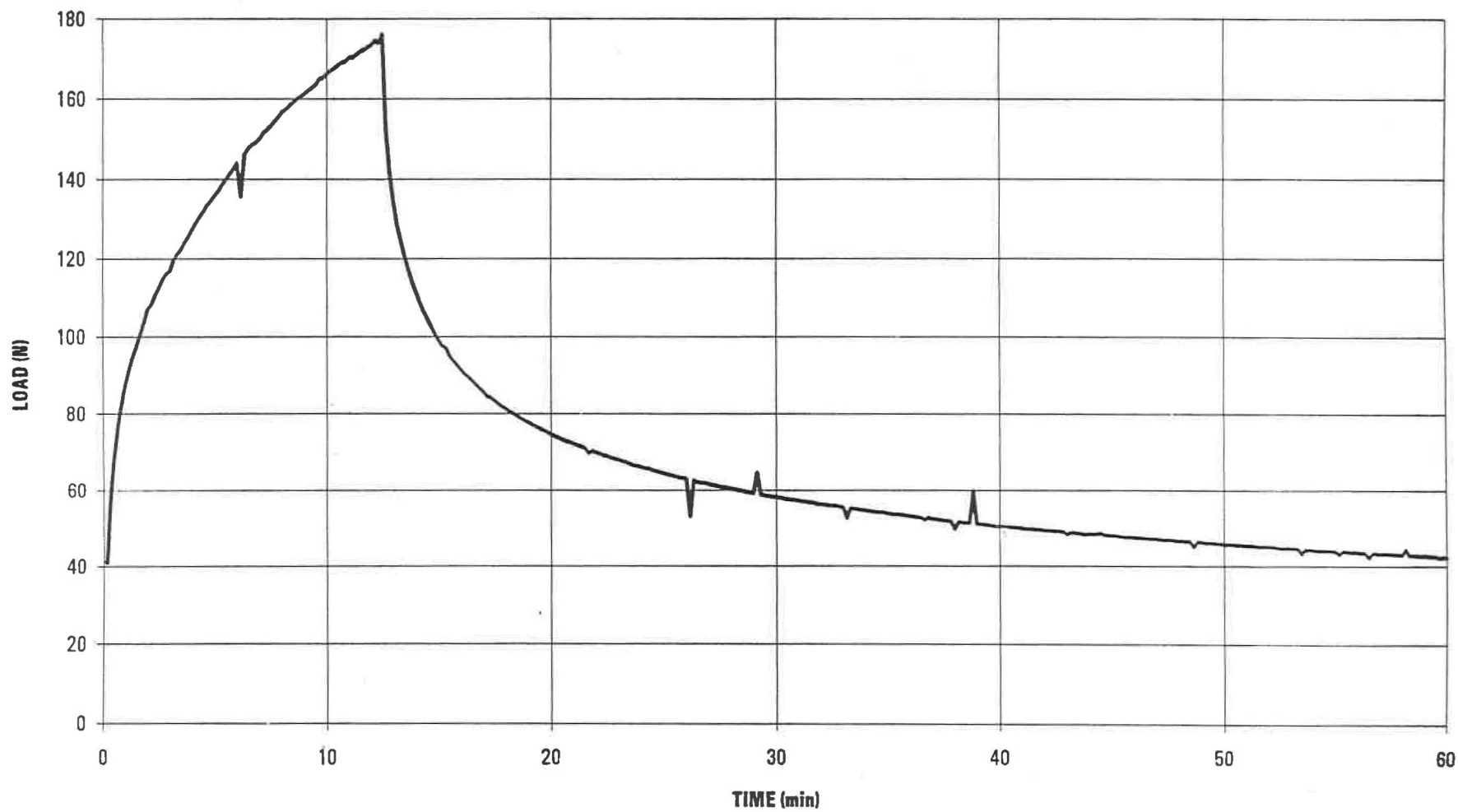


FIGURE 6. Stress Relaxation at -30°C, Sample 2382

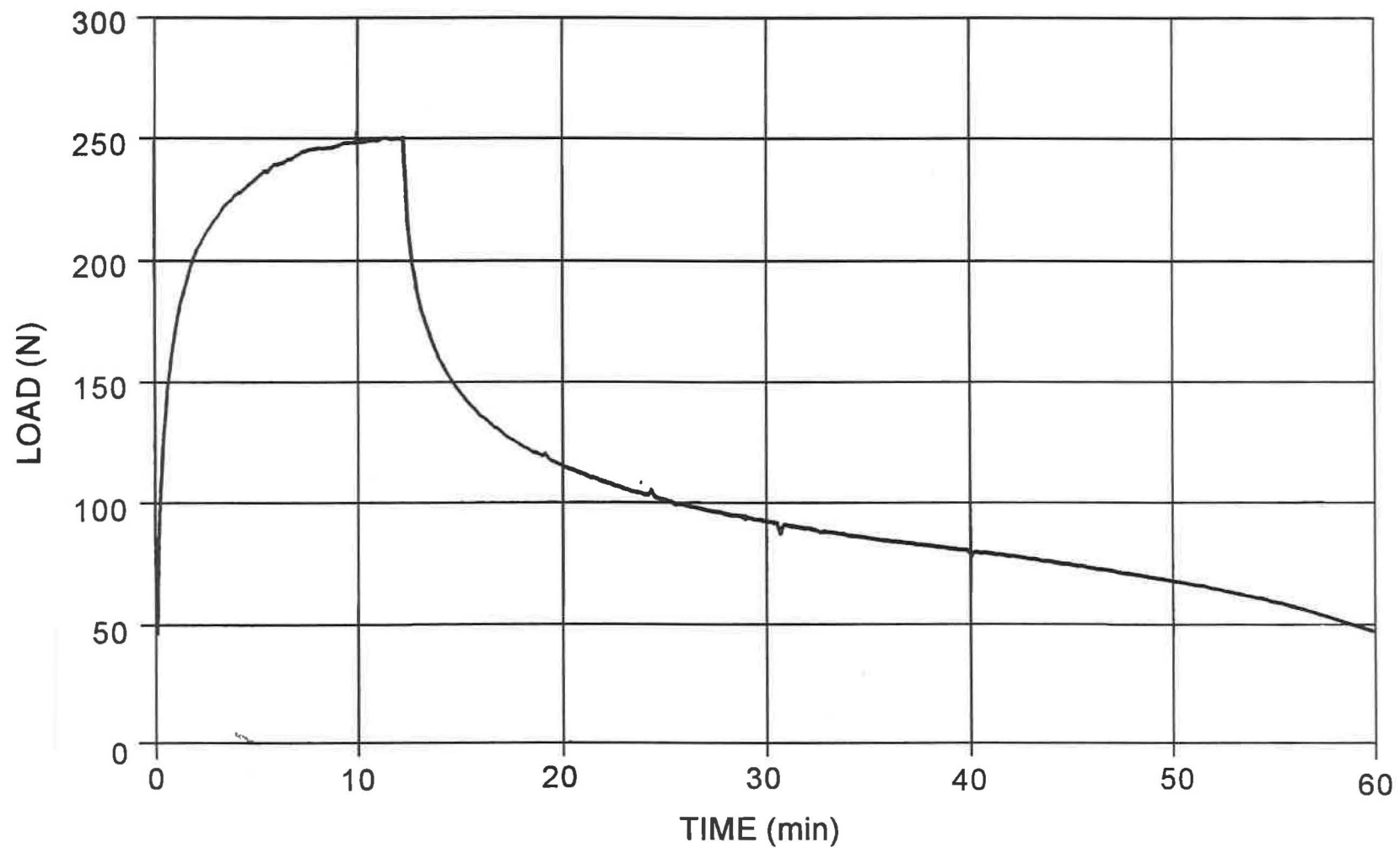


FIGURE 7. Stress Relaxation at -30°C, Sample 2383

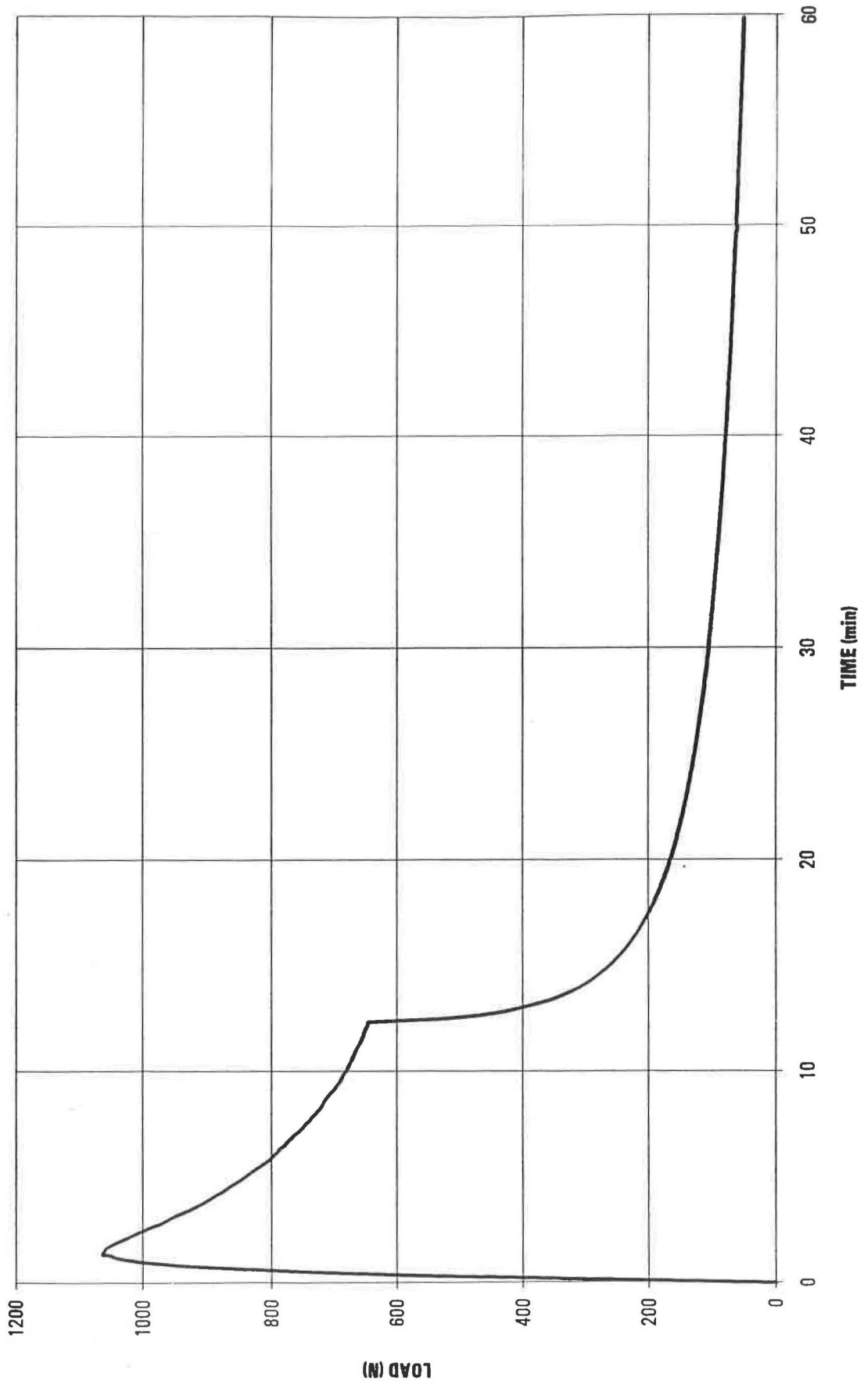


FIGURE 8. Stress Relaxation at -30°C, Sample 2384

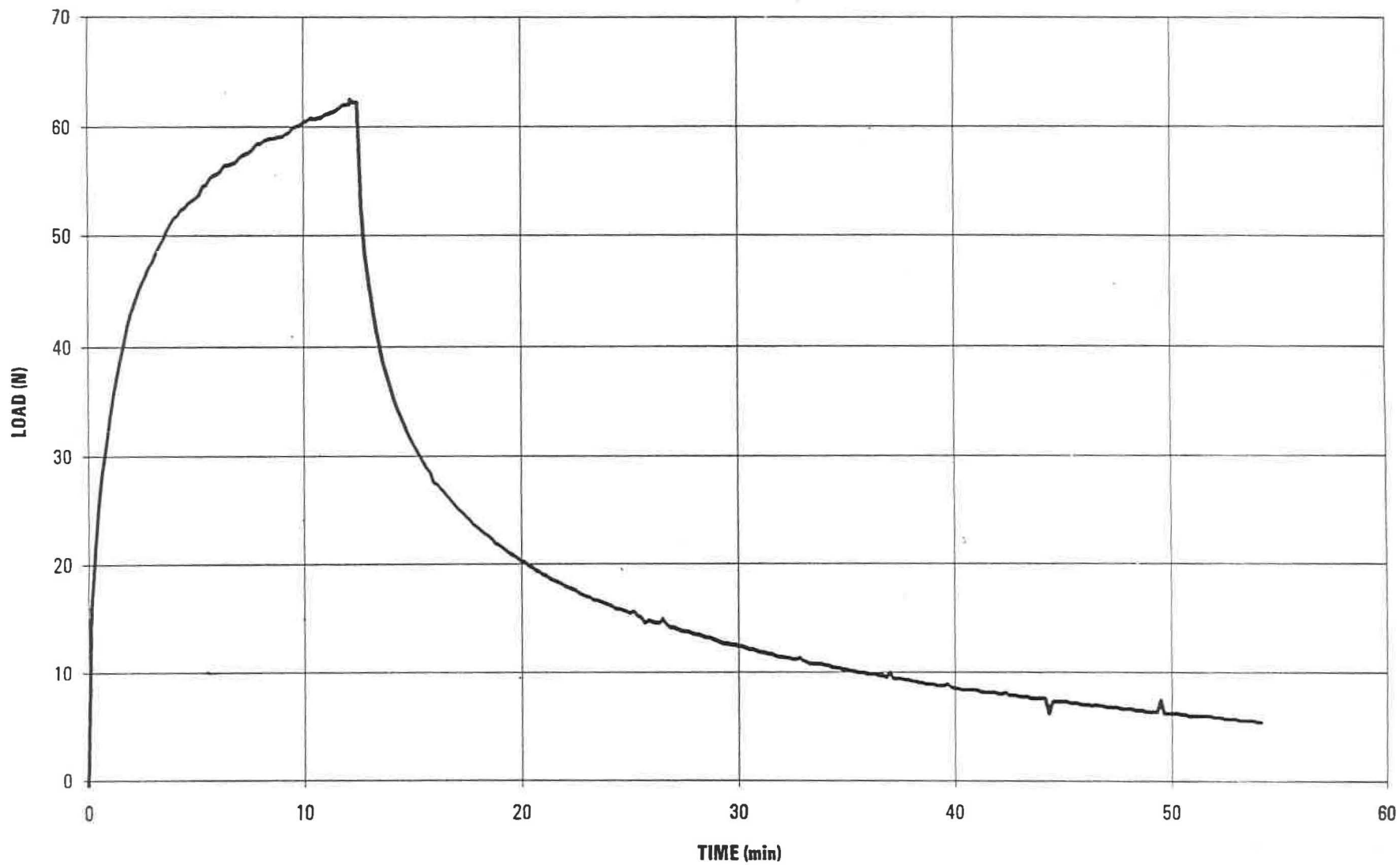


FIGURE 9. Stress Relaxation at -30°C, Sample 2385

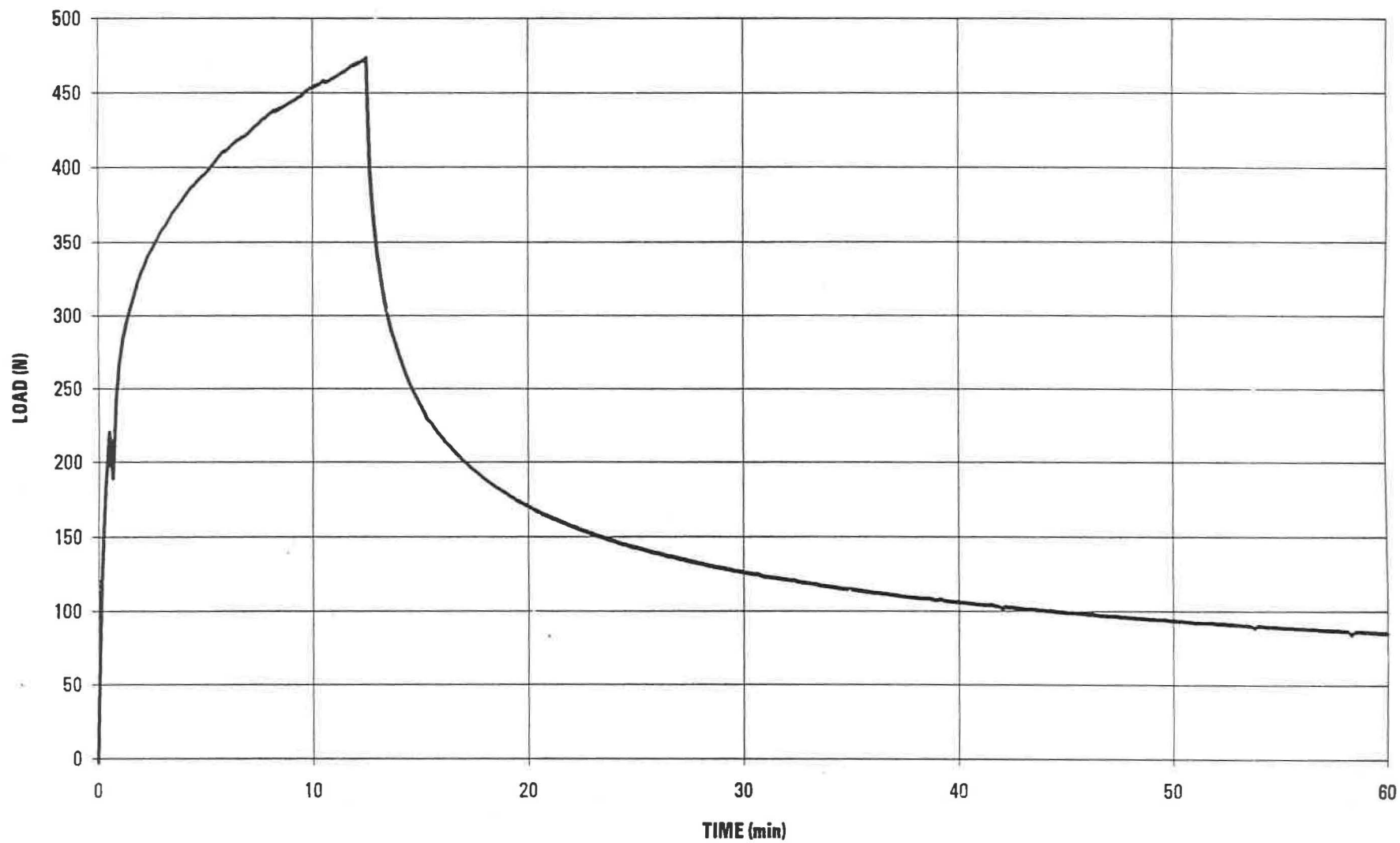


FIGURE 10. Stress Relaxation at -30°C, Sample 2386

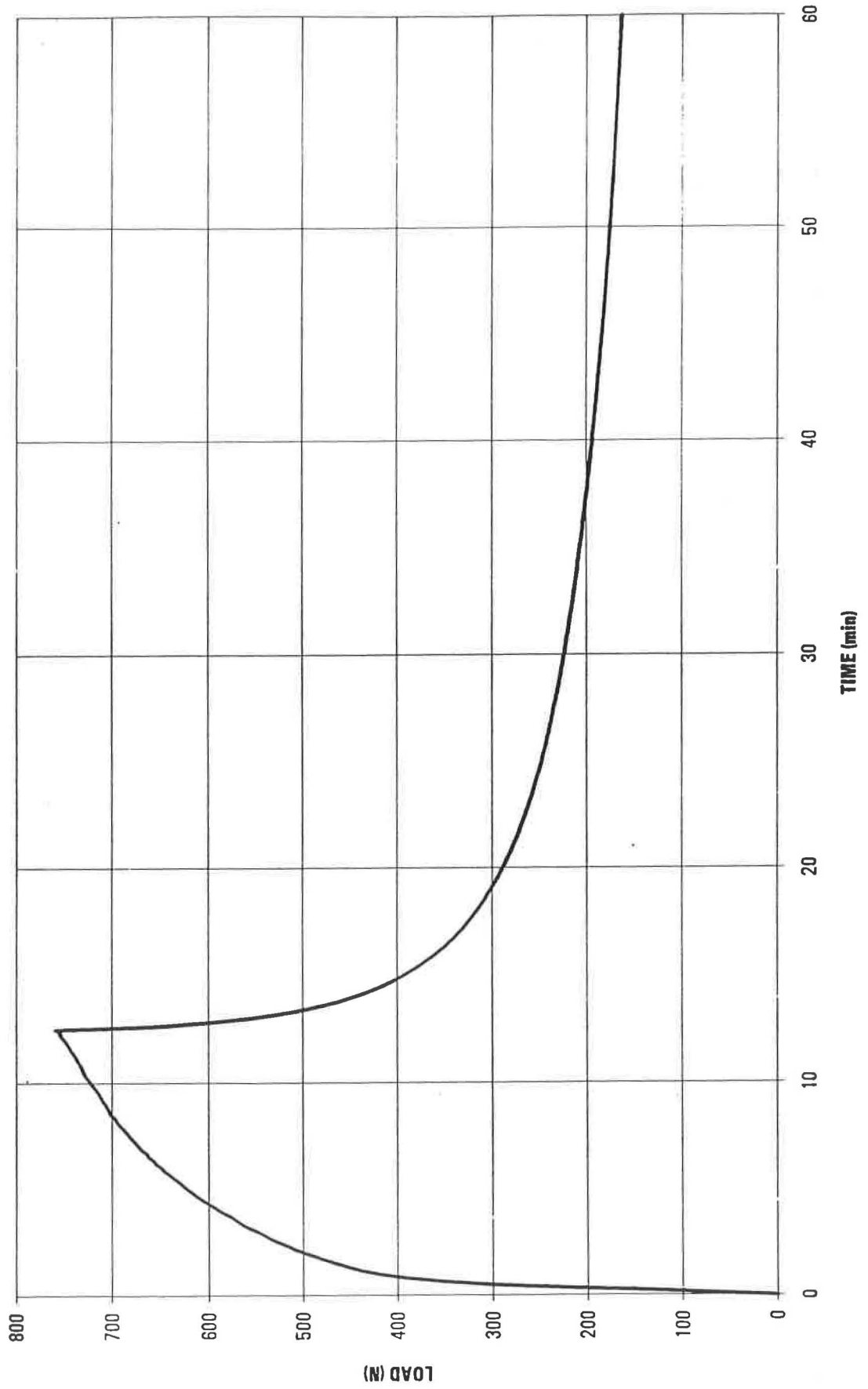


FIGURE 11. Stress Relaxation at -30°C, Sample 2387

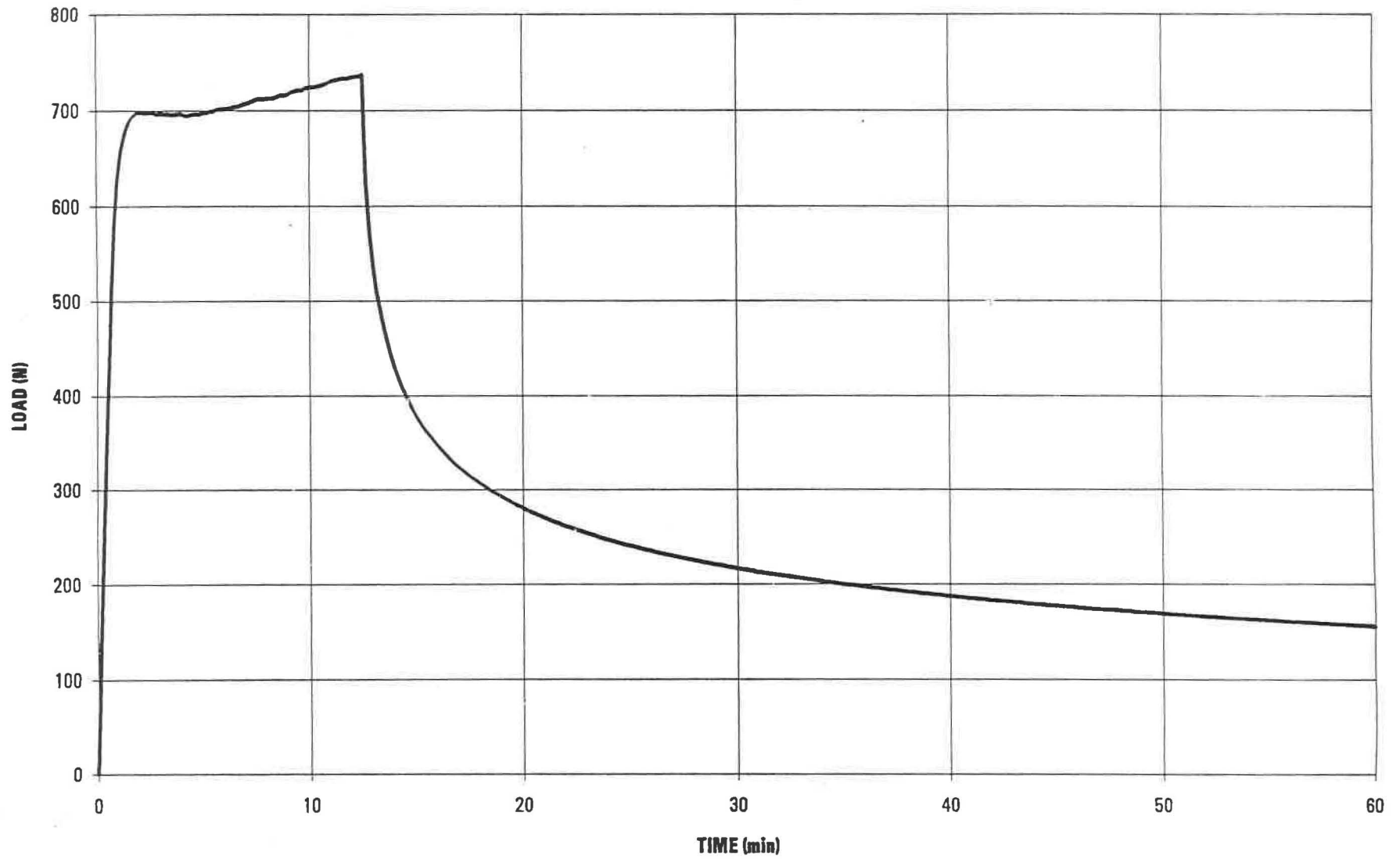


FIGURE 12. Stress Relaxation at -30°C, Sample 2388

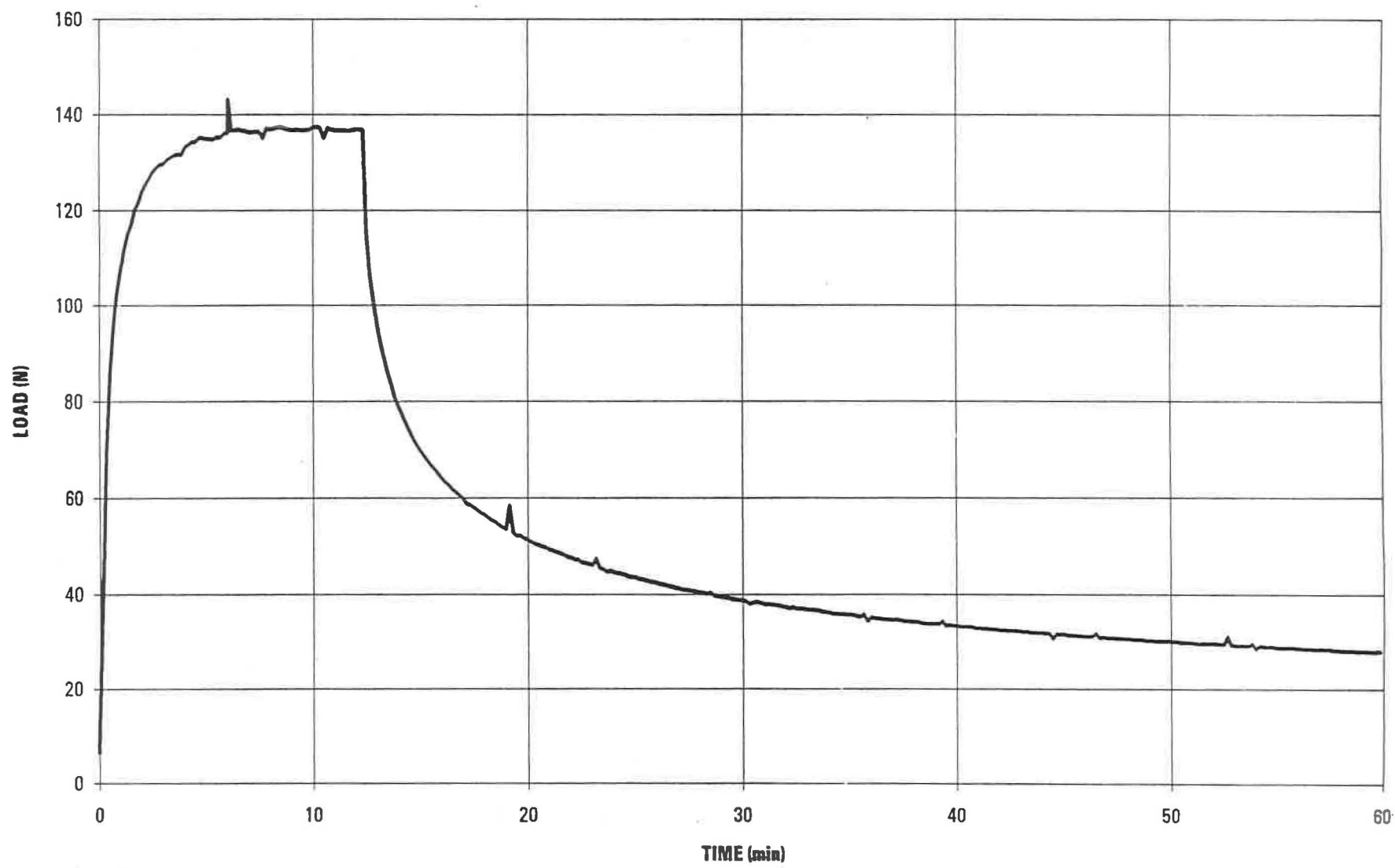


FIGURE 13. Stress Relaxation at -30°C, Sample 2389

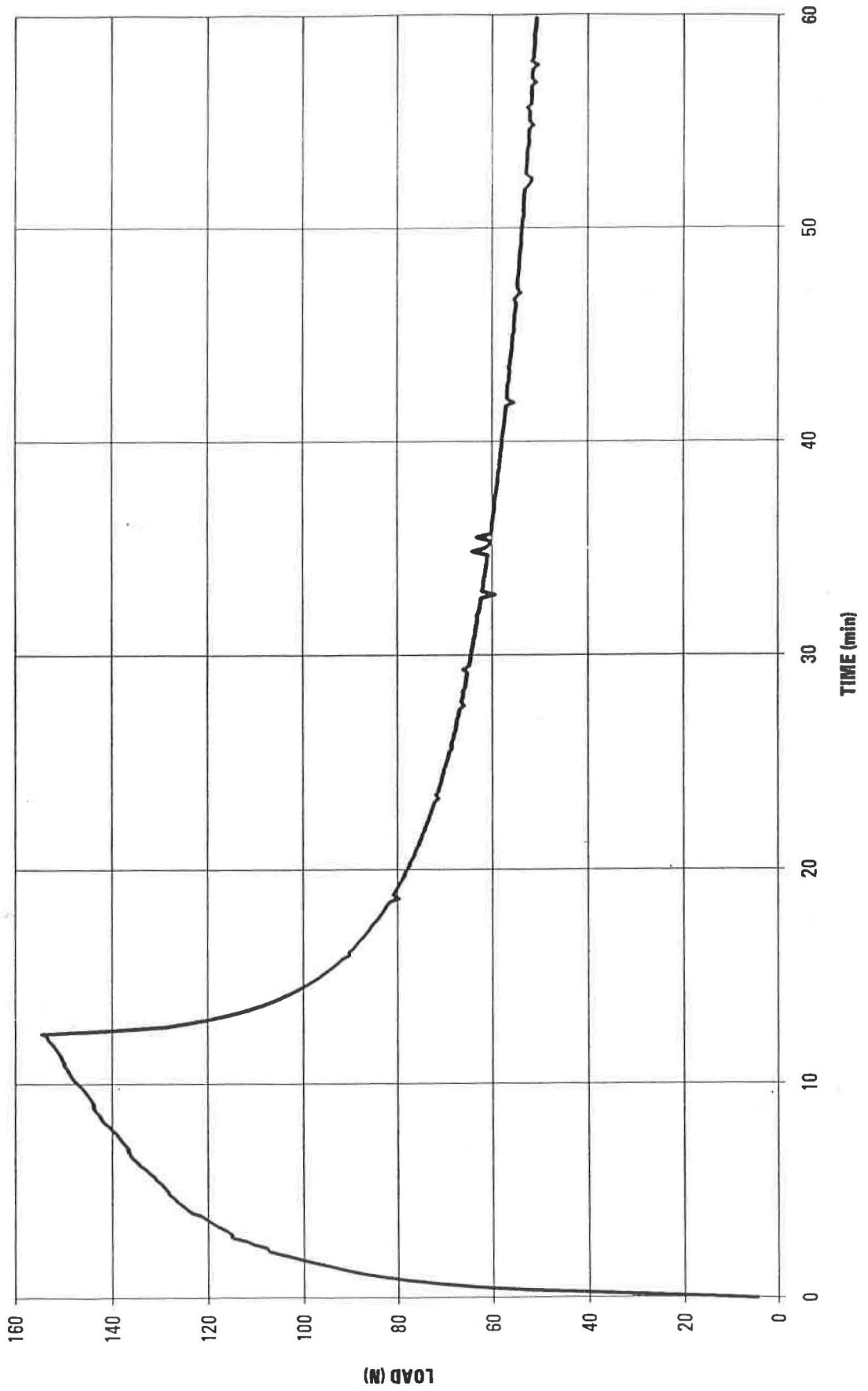


FIGURE 14. Stress Relaxation at -30°C, Sample 2390

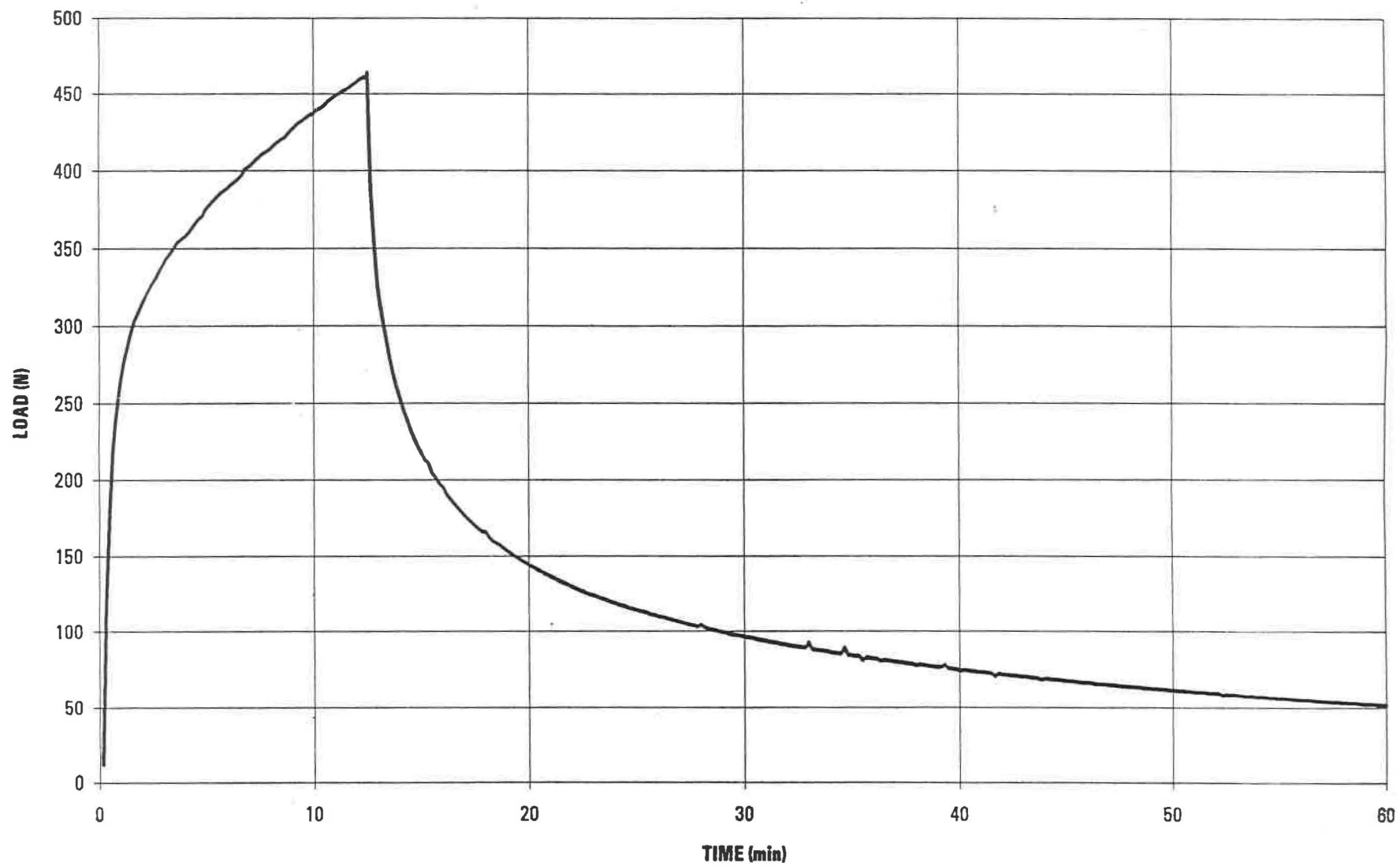


FIGURE 15. Stress Relaxation at -30°C, Sample 2448

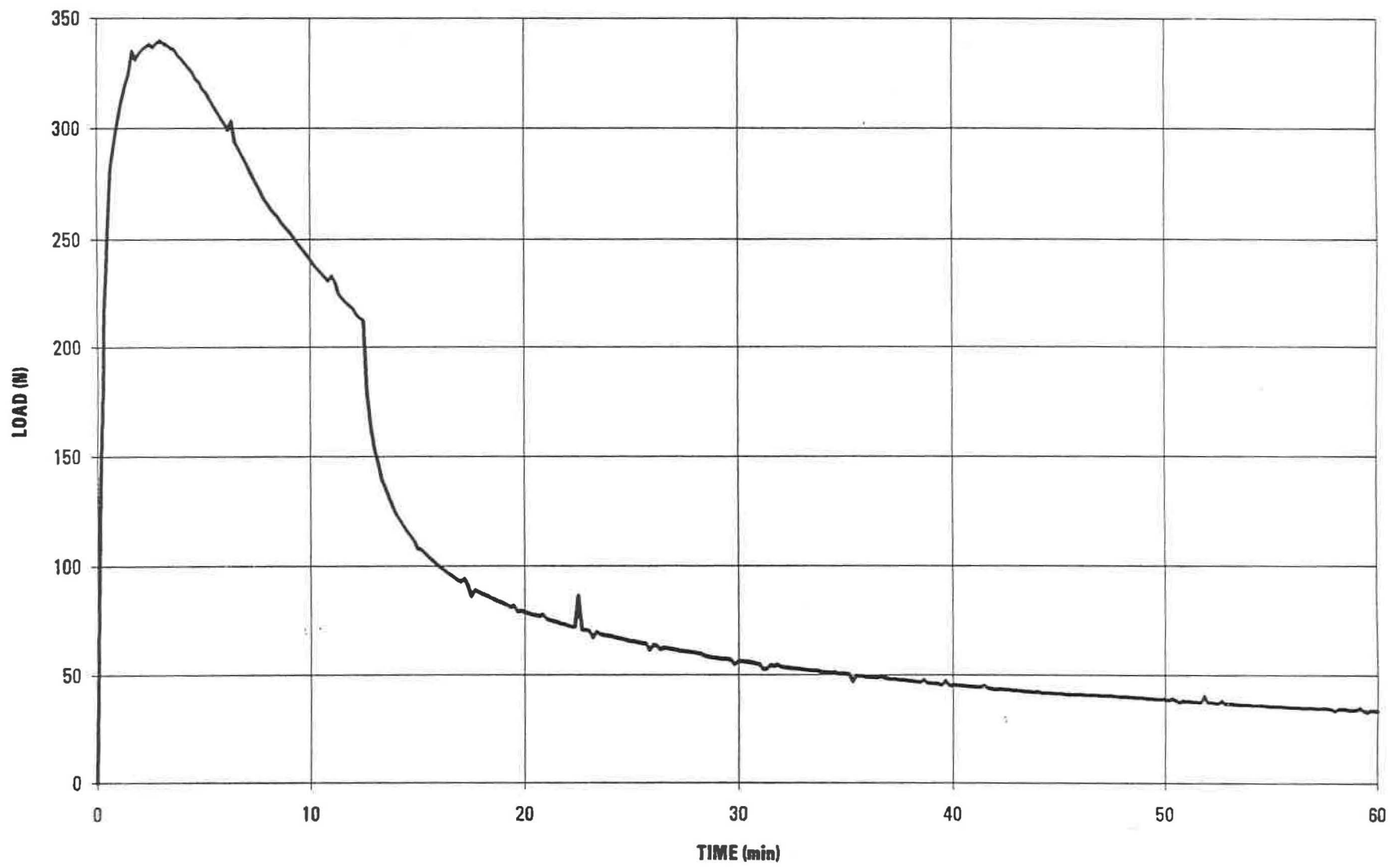


FIGURE 16. Stress Relaxation at -30°C, Sample 2449

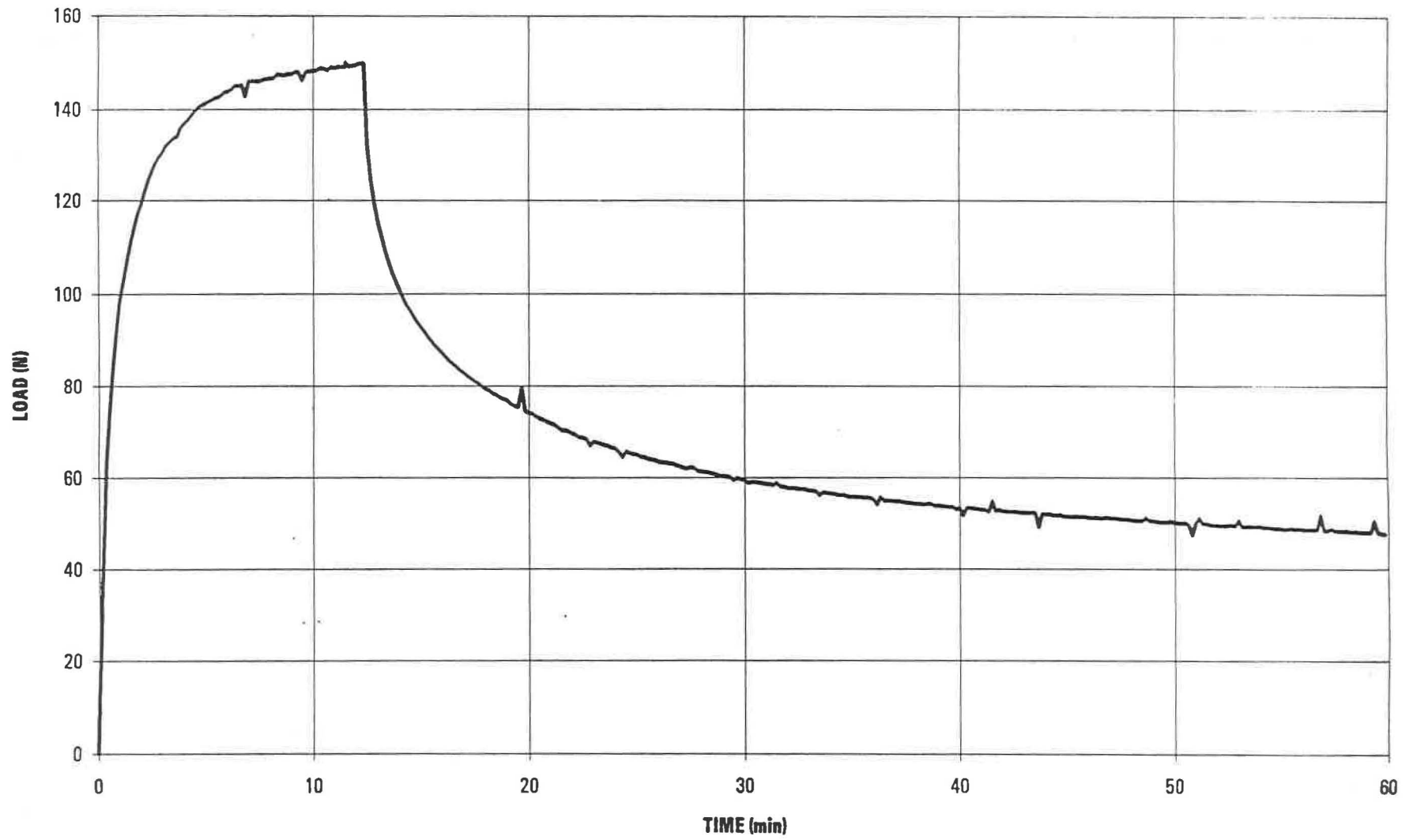


FIGURE 17. Stress Relaxation at -30°C, Sample 2450

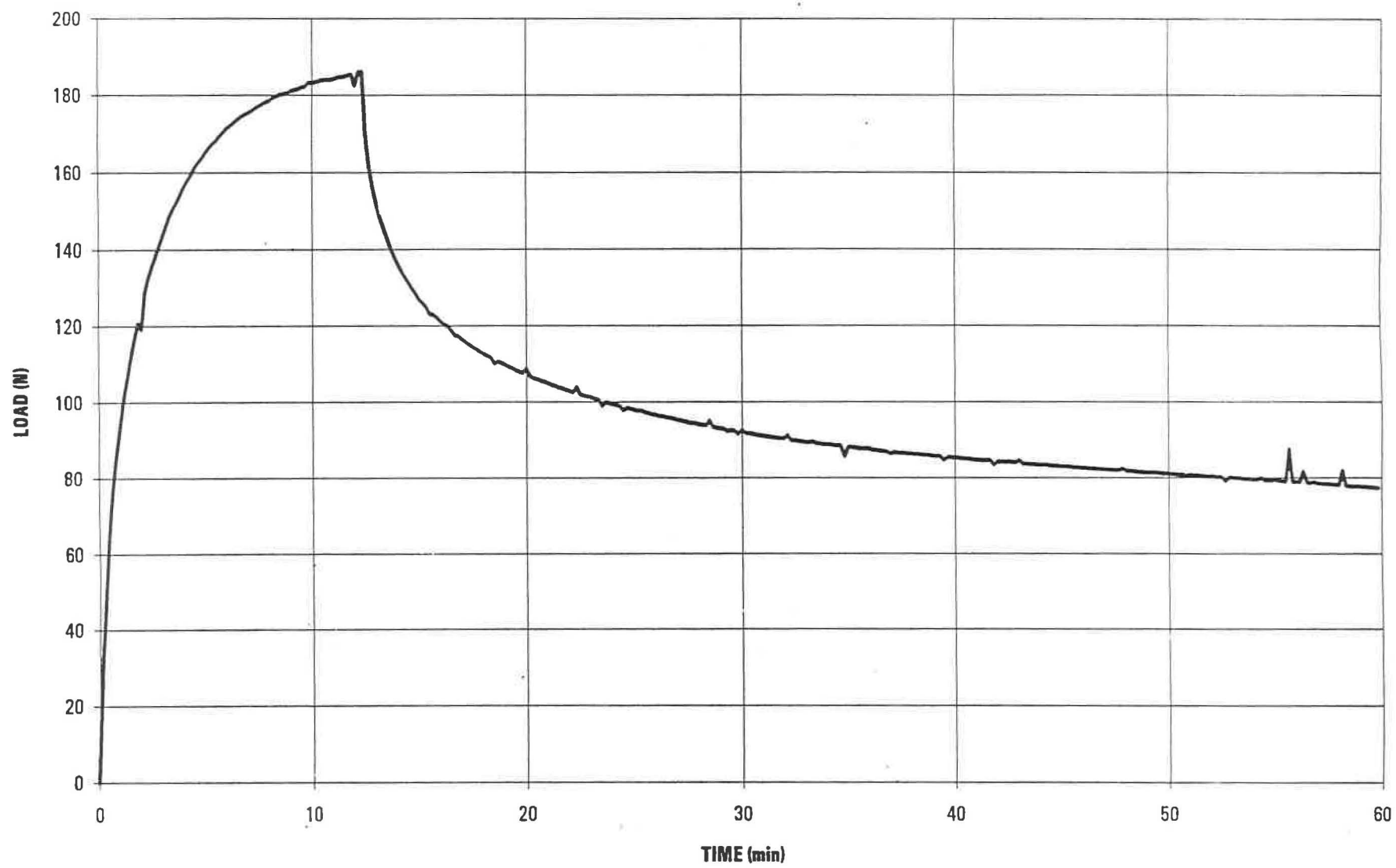
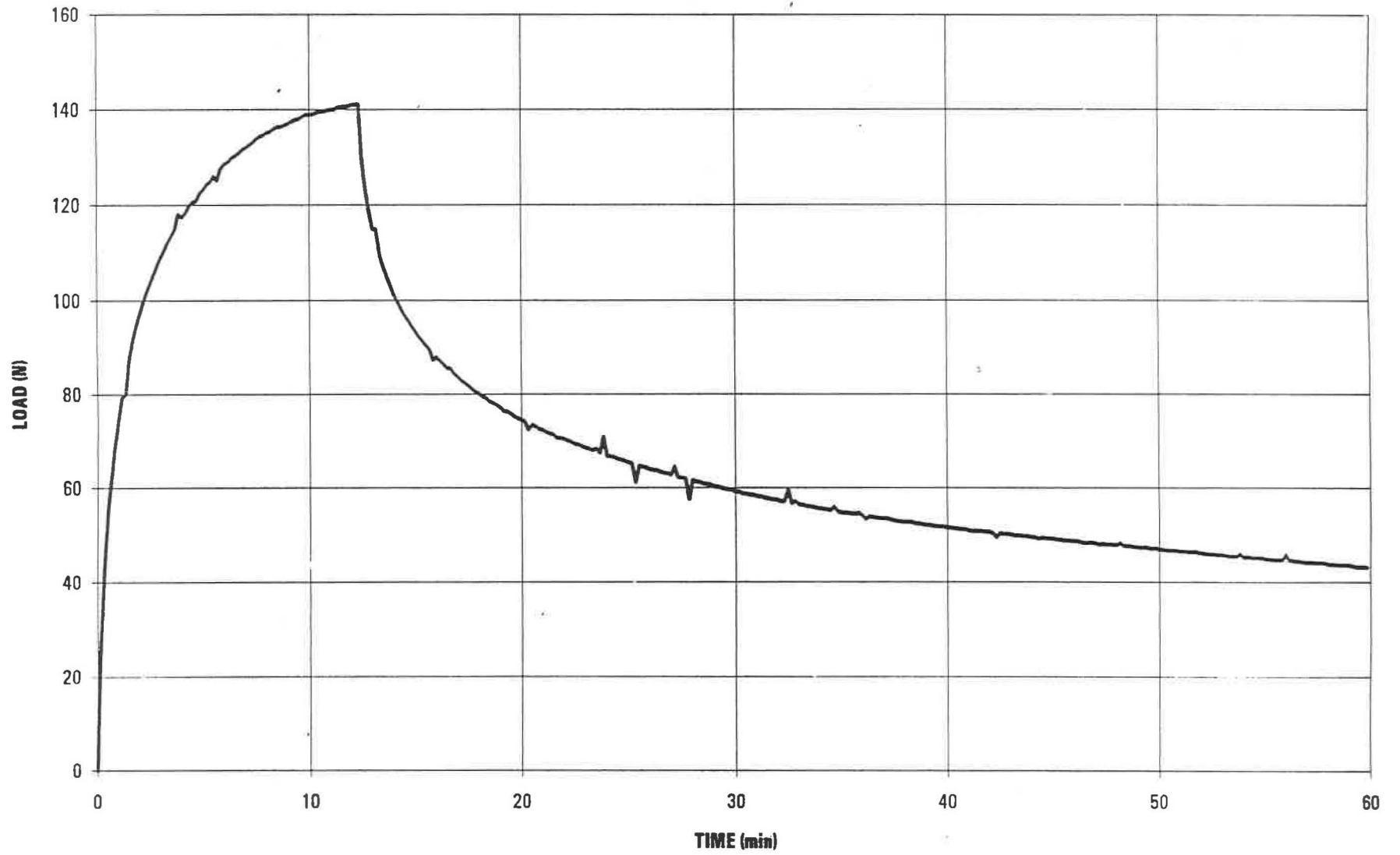


FIGURE 18. Stress Relaxation at -30°C, Sample 2451



Adhesion Test Using T&T System - TAC Proposal for C&J Test

Test Type:	Tensile	Instron Corporation
Sample Identification:		Series IX Automated Materials Testing System 1.02C
Interface Type:	4200 Series	Test Date: 19 Sep 1994
Machine Parameters of Test:		Sample Type: ASTM
Sample rate (pts/sec):	5.00	Humidity (%): 50
Crosshead Speed (mm/min):	10.000	Temperature (deg. C): -29

Sample Description: 2379

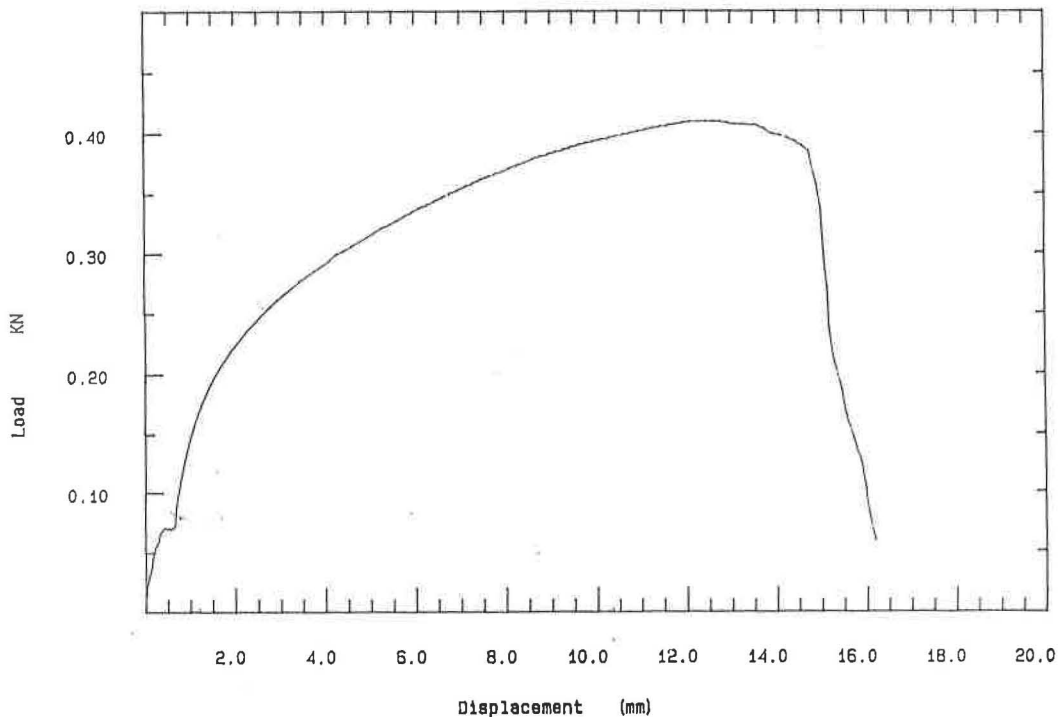
Dimensions:

	<u>Spec. 1</u>	<u>Spec. 2</u>	<u>Spec. 3</u>	<u>Spec. 4</u>
Diameter (mm)	25.400	25.400	25.400	25.400
Spec gauge len (mm)	26.049	25.362	25.467	25.483
Grip distance (mm)	125.000	125.000	125.000	125.000

Out of 4 specimens, 1 excluded.

Specimen No.	Toughness (J)	Peak Load (N)	Ratio of Toughness	Displacement at Max. Load (mm)	Maximum Displacement (mm)
1	4.745	421.7	.0113	10.400	15.08
2	5.100	411.4	.0124	12.660	16.23
Excluded	6.772	427.9	.0158	17.360	19.33
4	4.643	401.6	.0116	6.597	13.98
Mean	4.829	411.6	.0117	9.886	15.10
Std. Deviation	.240	10.1	.0006	3.063	1.13

FIGURE 19. Tensile Adhesion at -30C, Sample 2379



Adhesion Test Using T&T System - TAC Proposal for C&J Test

Test Type:	Tensile	Instron Corporation
Sample Identification:		Series IX Automated Materials Testing System 1.02C
Interface Type:	4200 Series	Test Date: 19 Sep 1994
Machine Parameters of Test:		Sample Type: ASTM
Sample rate (pts/sec):	5.00	Humidity (%): 50
Crosshead Speed (mm/min):	10.000	Temperature (deg. C): -29

Sample Description: 2380

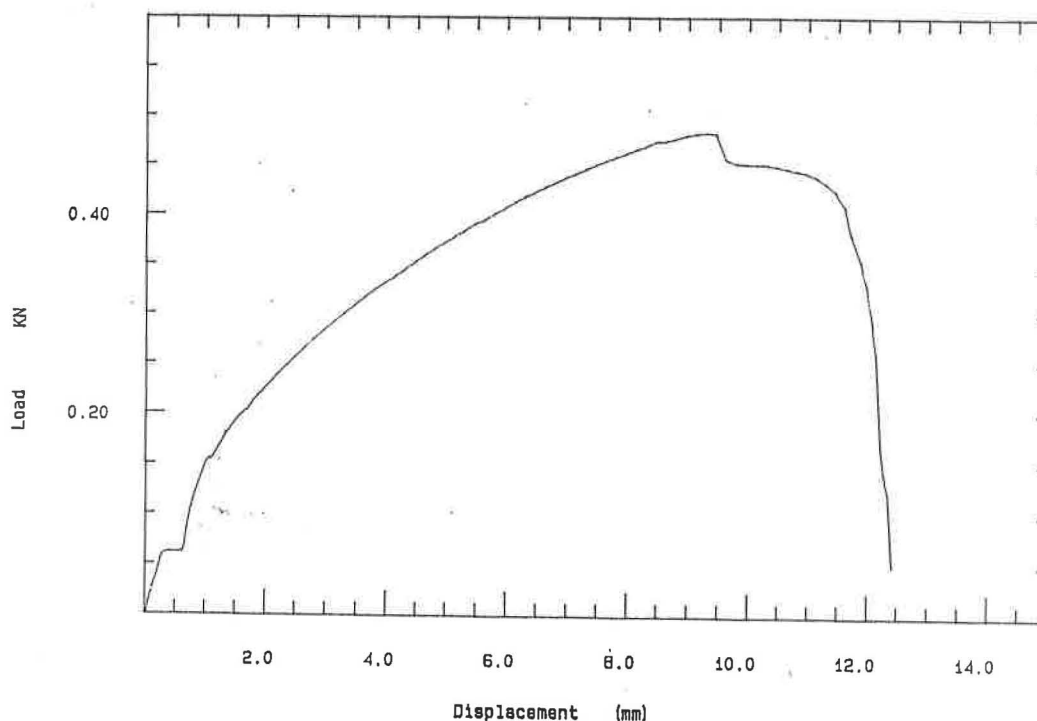
Dimensions:

	<u>Spec. 1</u>	<u>Spec. 2</u>	<u>Spec. 3</u>
Diameter (mm)	25.400	25.400	25.400
Spec gauge len (mm)	27.523	25.429	26.566
Grip distance (mm)	125.000	125.000	125.000

Out of 3 specimens, 0 excluded.

Specimen No.	Toughness (J)	Peak Load (N)	Ratio of Toughness	Displacement at Max. Load (mm)	Maximum Displacement (mm)
1	3.262	558.9	.0058	7.927	8.098
2	4.328	484.8	.0089	9.171	12.400
3	5.000	524.8	.0095	11.400	13.660
Mean	4.196	522.8	.0081	9.500	11.380
Std. Deviation	.876	37.1	.0020	1.762	2.915

FIGURE 20. Tensile Adhesion at -30C, Sample 2380



Adhesion Test Using T&T System - TAC Proposal for C&J Test

Test Type:	Tensile	Instron Corporation
Sample Identification:	AT2381A3	Series IX Automated Materials Testing System 1.02C
Interface Type:	4200 Series	Test Date: 19 Sep 1994
Machine Parameters of Test:		Sample Type: ASTM
Sample rate (pts/sec):	5.00	Humidity (%): 50
Crosshead Speed (mm/min):	10.000	Temperature (deg. C): -29

Sample Description: 2381

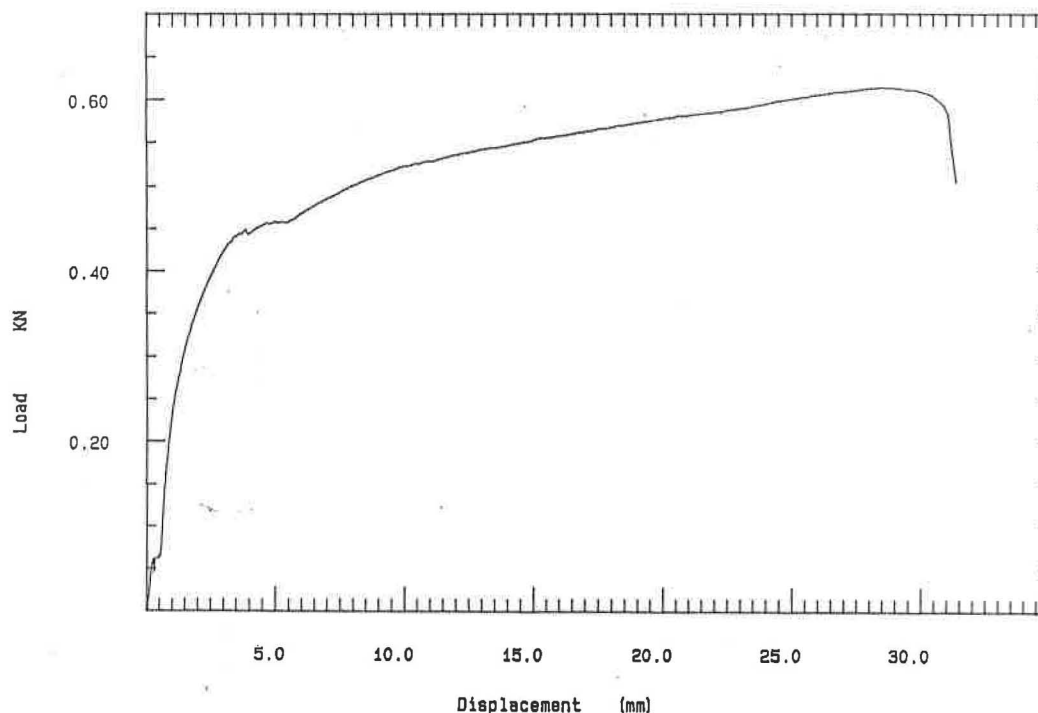
Dimensions:

	<u>Spec. 1</u>	<u>Spec. 2</u>	<u>Spec.3</u>	<u>Spec. 4</u>
Diameter (mm)	25.400	25.400	25.400	25.400
Spec gauge len (mm)	25.425	26.926	25.570	26.335
Grip distance (mm)	125.000	125.000	125.000	125.000

Out of 4 specimens, 0 excluded.

Specimen No.	Toughness (J)	Peak Load (N)	Ratio of Toughness	Displacement at Max. Load (mm)	Maximum Displacement (mm)
1	16.37	615.0	.0266	28.63	31.37
2	16.94	640.5	.0265	31.44	33.03
3	14.32	636.8	.0225	27.67	28.32
4	12.49	593.6	.0210	23.10	27.52
Mean	15.03	621.5	.0241	27.71	30.06
Std. Deviation	2.03	21.7	.0028	3.47	2.58

FIGURE 21. Tensile Adhesion at -30C, Sample 2381



Adhesion Test Using T&T System - TAC Proposal for C&J Test

Test Type:	Tensile	Instron Corporation
Sample Identification:		Series IX Automated Materials Testing System 1.02C
Interface Type:	4200 Series	Test Date: 21 Sep 1994
Machine Parameters of Test:		Sample Type: ASTM
Sample rate (pts/sec):	5.00	Humidity (%): 50
Crosshead Speed (mm/min):	10.000	Temperature (deg. C): -29

Sample Description: 2382

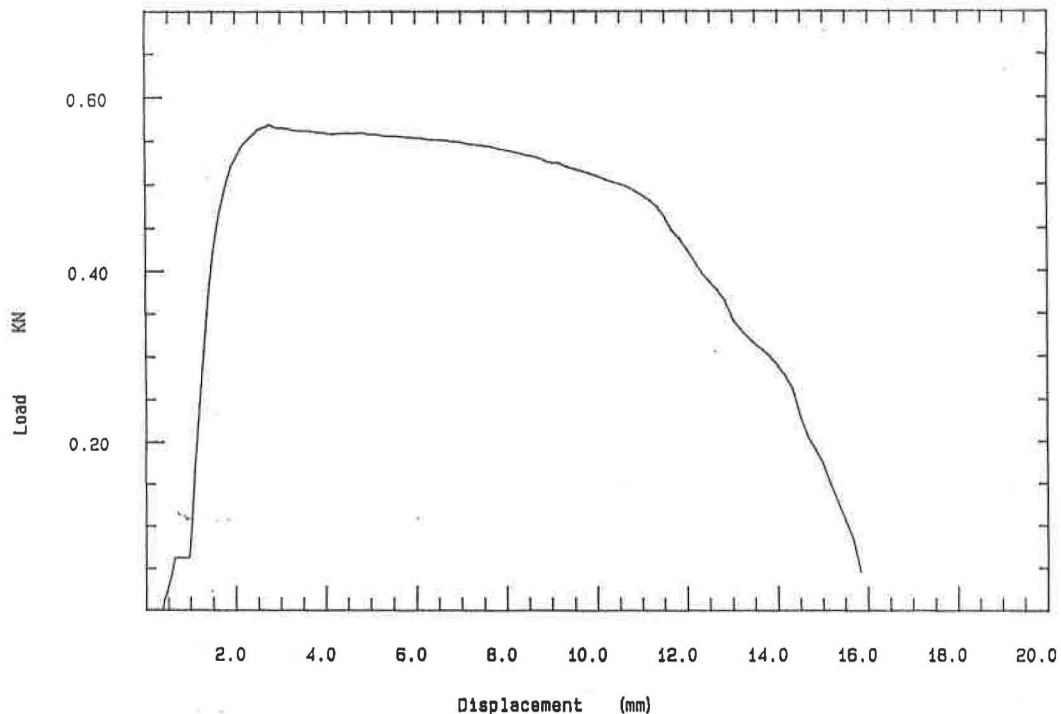
Dimensions:

	<u>Spec. 1</u>	<u>Spec. 2</u>	<u>Spec. 3</u>	<u>Spec. 4</u>
Diameter (mm)	25.400	25.400	25.400	25.400
Spec gauge len (mm)	25.776	25.392	26.081	29.062
Grip distance (mm)	125.000	125.000	125.000	125.000

Out of 4 specimens, 1 excluded.

Specimen No.	Toughness (J)	Peak Load (N)	Ratio of Toughness	Displacement at Max. Load (mm)	Maximum Displacement (mm)
1	6.765	568.6	.0119	2.354	15.45
Excluded	3.397	521.9	.0065	1.978	10.96
3	7.215	594.9	.0121	2.129	14.61
4	5.610	605.6	.0093	3.248	12.53
Mean	6.530	589.7	.0111	2.577	14.20
Std. Deviation	.828	19.0	.0016	.591	1.51

FIGURE 22. Tensile Adhesion at -30C, Sample 2382



Adhesion Test Using T&T System - TAC Proposal for C&J Test

Test Type:	Tensile	Instron Corporation
Sample Identification:		Series IX Automated Materials Testing System 1.02C
Interface Type:	4200 Series	Test Date: 21 Sep 1994
Machine Parameters of Test:		Sample Type: ASTM
Sample rate (pts/sec):	5.00	Humidity (%): 50
Crosshead Speed (mm/min):	10.000	Temperature (deg. C): -29

Sample Description: 2383

Dimensions:

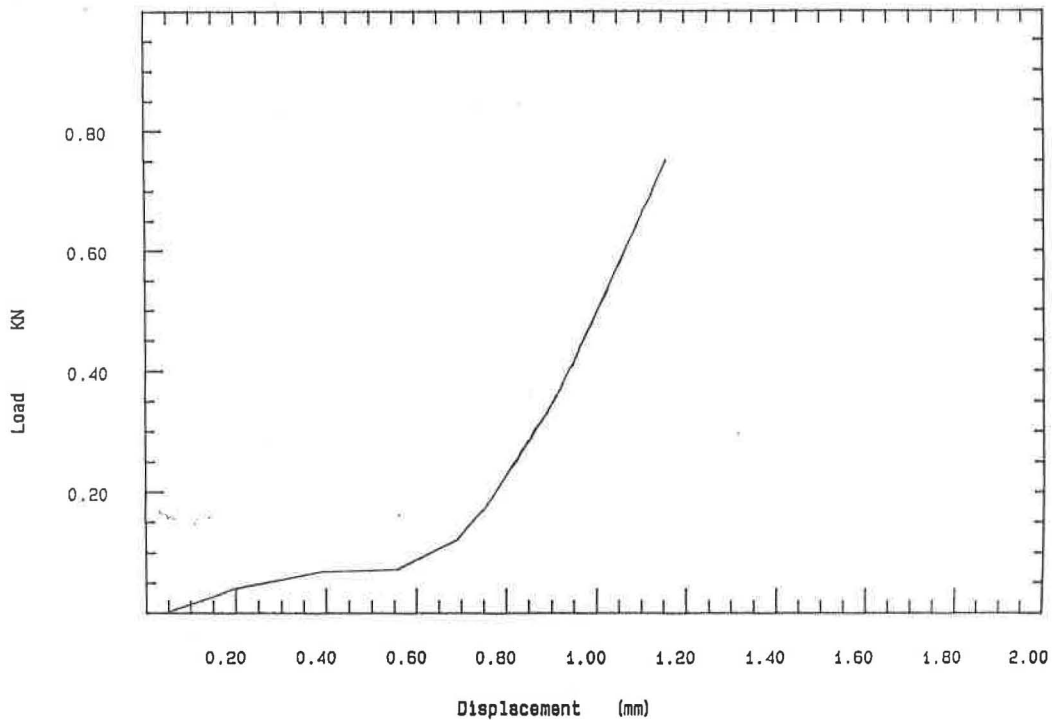
	<u>Spec. 1</u>	<u>Spec. 2</u>	<u>Spec.3</u>	<u>Spec. 4</u>
Diameter (mm)	25.400	25.400	25.400	25.400
Spec gauge len (mm)	28.167	26.105	26.097	26.228
Grip distance (mm)	125.000	125.000	125.000	125.000

Out of 4 specimens, 1 excluded.

Sample Comments: Sample broke, did NOT detach from brick

Specimen No.	Toughness (J)	Peak Load (N)	Ratio of Toughness	Displacement at Max. Load (mm)	Maximum Displacement (mm)
Excluded	.3452	1111.0	.0003	.4833	.4624
2	.2261	767.8	.0003	.4752	.4535
3	.2024	796.2	.0003	.4131	.4254
4	.1553	681.1	.0002	.3424	.3303
Mean	.1946	748.4	.0003	.4102	.4031
Std. Deviation	.0361	60.0	.0000	.0665	.0646

FIGURE 23. Tensile Adhesion at -30C, Sample 2383



Adhesion Test Using T&T System - TAC Proposal for C&J Test

Test Type:	Tensile	Instron Corporation
Sample Identification:	T2384	Series IX Automated Materials Testing System 1.02C
Interface Type:	4200 Series	Test Date: 21 Sep 1994
Machine Parameters of Test:		Sample Type: ASTM
Sample rate (pts/sec):	5.00	Humidity (%): 50
Crosshead Speed (mm/min):	10.000	Temperature (deg. C): -29

Sample Description: 2384

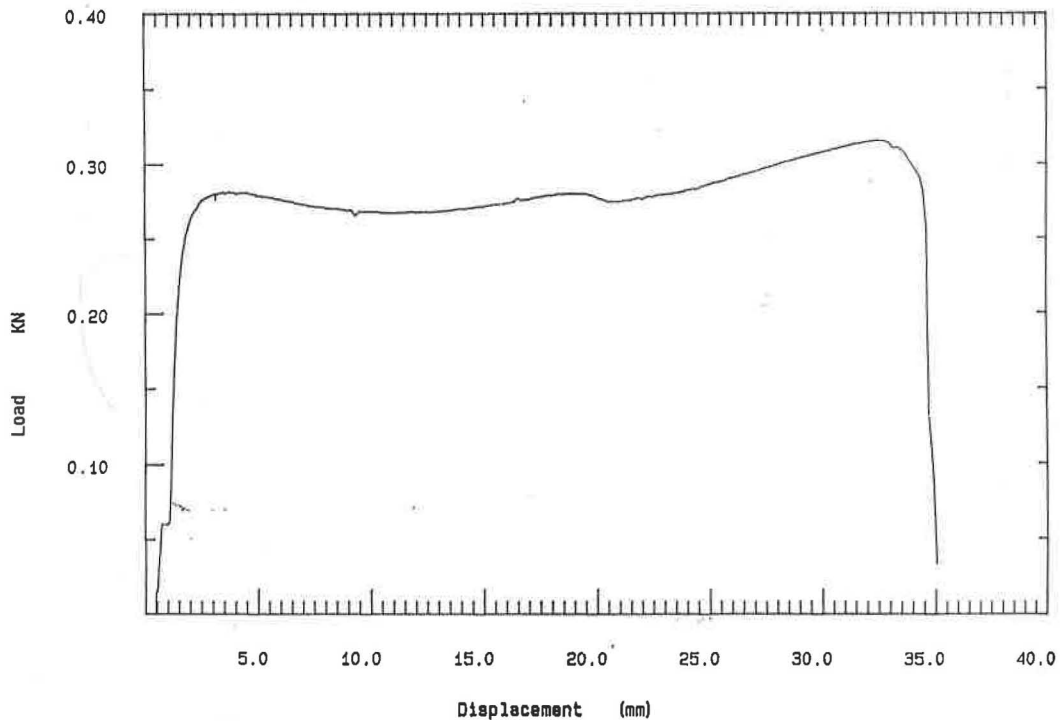
Dimensions:

	<u>Spec. 1</u>	<u>Spec. 2</u>	<u>Spec. 3</u>	<u>Spec. 4</u>
Diameter (mm)	25.400	25.400	25.400	25.400
Spec gauge len (mm)	25.851	25.492	25.409	25.660
Grip distance (mm)	125.000	125.000	125.000	125.000

Out of 4 specimens, 0 excluded.

Specimen No.	Toughness (J)	Peak Load (N)	Ratio of Toughness	Displacement at Max. Load (mm)	Maximum Displacement (mm)
1	9.495	316.6	.0300	32.060	34.58
2	7.976	313.8	.0254	2.858	28.00
3	6.341	350.6	.0181	2.691	22.82
4	10.250	327.2	.0313	34.190	35.97
Mean	8.516	327.0	.0262	17.950	30.34
Std. Deviation	1.732	16.7	.0060	17.540	6.10

FIGURE 24. Tensile Adhesion at -30C, Sample 2384



Adhesion Test Using T&T System - TAC Proposal for C&J Test

Test Type:	Tensile	Instron Corporation
Sample Identification:		Series IX Automated Materials Testing System 1.02C
Interface Type:	4200 Series	Test Date: 27 Sep 1994
Machine Parameters of Test:		Sample Type: ASTM
Sample rate (pts/sec):	5.00	Humidity (%): 50
Crosshead Speed (mm/min):	10.000	Temperature (deg. C): -29

Sample Description: 2385

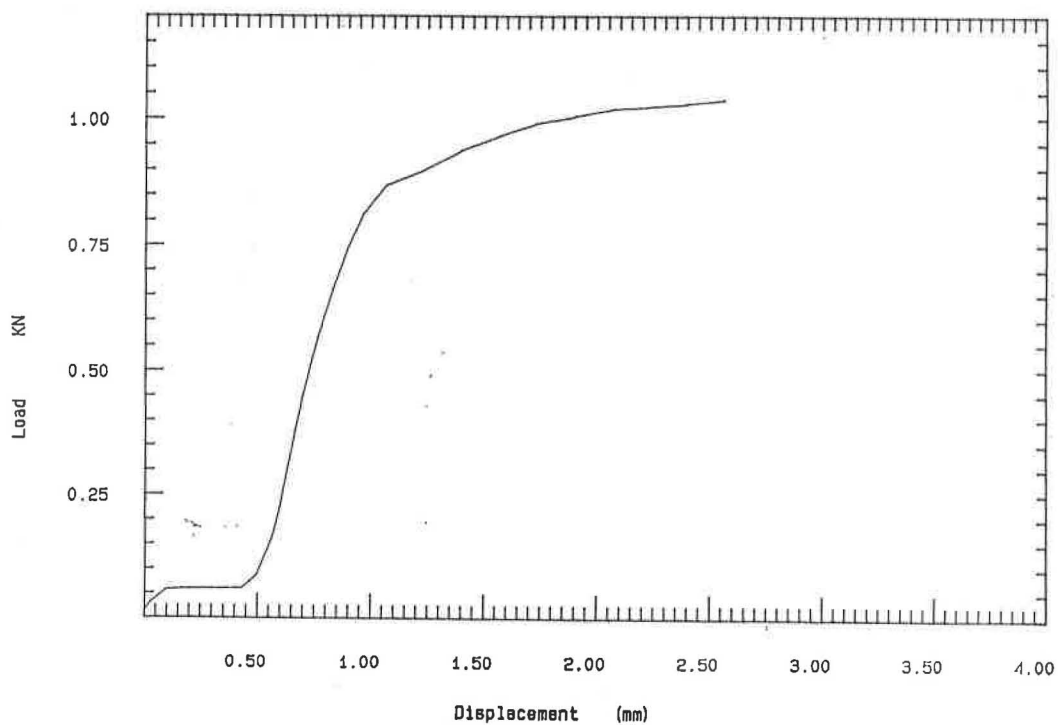
Dimensions:

	<u>Spec. 1</u>	<u>Spec. 2</u>	<u>Spec. 3</u>	<u>Spec. 4</u>
Diameter (mm)	25.400	25.400	25.400	25.400
Spec gauge len (mm)	25.885	25.981	25.966	26.681
Grip distance (mm)	125.000	125.000	125.000	125.000

Out of 4 specimens, 1 excluded.

Specimen No.	Toughness (J)	Peak Load (N)	Ratio of Toughness	Displacement at Max. Load (mm)	Maximum Displacement (mm)
1	1.8090	1039.0	.0017	2.1050	2.0770
Excluded	1.0750	864.4	.0012	.8390	3.0760
3	.5478	973.2	.0006	1.0140	.8881
4	1.0460	1068.0	.0010	1.3890	1.3130
Mean	1.1340	1027.0	.0011	1.5030	1.4260
Std. Deviation	.6353	48.6	.0006	.5542	.6024

FIGURE 25. Tensile Adhesion at -30C, Sample 2385



Adhesion Test Using T&T System - TAC Proposal for C&J Test

Test Type:	Tensile	Instron Corporation
Sample Identification:	21230002	Series IX Automated Materials Testing System 1.02C
Interface Type:	4200 Series	Test Date: 27 Sep 1994
Machine Parameters of Test:		Sample Type: ASTM
Sample rate (pts/sec):	5.00	Humidity (%): 50
Crosshead Speed (mm/min):	10.000	Temperature (deg. C): -29

Sample Description: 2386

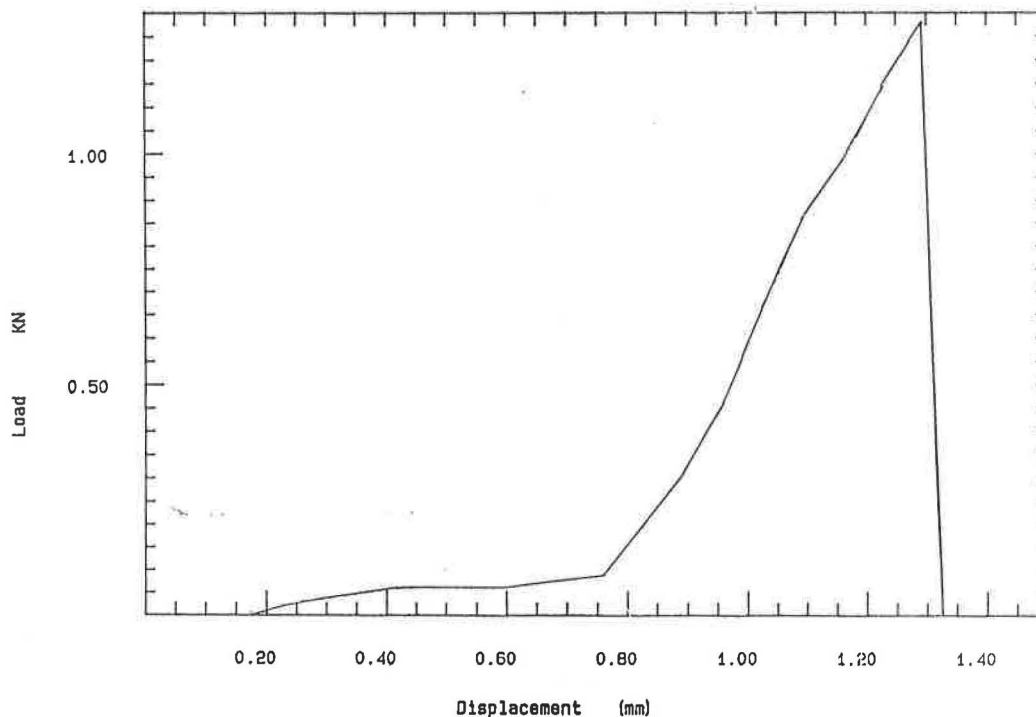
Dimensions:

	<u>Spec. 1</u>	<u>Spec. 2</u>	<u>Spec. 3</u>	<u>Spec. 4</u>
Diameter (mm)	25.400	25.400	25.400	25.400
Spec gauge len (mm)	26.066	26.211	26.065	25.934
Grip distance (mm)	125.000	125.000	125.000	125.000

Out of 4 specimens, 0 excluded.

Specimen No.	Toughness (J)	Peak Load (N)	Ratio of Toughness	Displacement at Max. Load (mm)	Maximum Displacement (mm)
1	.1874	823.6	.0002	.3345	.3231
2	.3860	1287.0	.0003	.4890	.5156
3	.1726	801.1	.0002	.3553	.3260
4	.2547	1179.0	.0002	.3560	.3539
Mean	.2502	1023.0	.0002	.3837	.3796
Std. Deviation	.0974	247.0	.0000	.0709	.0917

FIGURE 26. Tensile Adhesion at -30C, Sample 2386



Adhesion Test Using T&T System - TAC Proposal for C&J Test

Test Type:	Tensile	Instron Corporation
Sample Identification:	AT238723	Series IX Automated Materials Testing System 1.02C
Interface Type:	4200 Series	Test Date: 27 Sep 1994
Machine Parameters of Test:		Sample Type: ASTM
Sample rate (pts/sec):	5.00	Humidity (%): 50
Crosshead Speed (mm/min):	10.000	Temperature (deg. C): -29

Sample Description: 2387

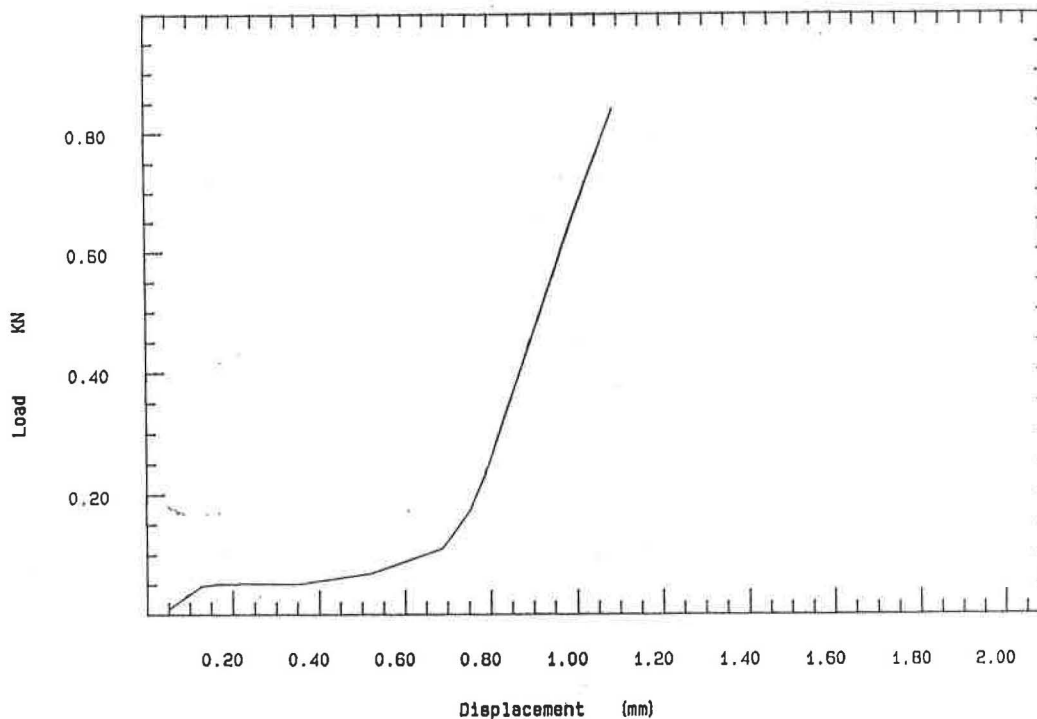
Dimensions:

	<u>Spec. 1</u>	<u>Spec. 2</u>	<u>Spec.3</u>	<u>Spec. 4</u>
Diameter (mm)	25.400	25.400	25.400	25.400
Spec gauge len (mm)	27.062	26.080	26.360	26.189
Grip distance (mm)	125.000	125.000	125.000	125.000

Out of 4 specimens, 0 excluded.

Specimen No.	Toughness (J)	Peak Load (N)	Ratio of Toughness	Displacement at Max. Load (mm)	Maximum Displacement (mm)
1	.1891	776.6	.0002	.3578	.3608
2	.2192	849.7	.0003	.4204	.4104
3	.2075	695.6	.0003	.4605	.4678
4	.2917	949.5	.0003	.5306	.5339
Mean	.2269	817.9	.0003	.4423	.4432
Std. Deviation	.0449	108.0	.0000	.0725	.0746

FIGURE 27. Tensile Adhesion at -30C, Sample 2387



Adhesion Test Using T&T System - TAC Proposal for C&J Test

Test Type:	Tensile	Instron Corporation
Sample Identification:		Series IX Automated Materials Testing System 1.02C
Interface Type:	4200 Series	Test Date: 23 Sep 1994
Machine Parameters of Test:		Sample Type: ASTM
Sample rate (pts/sec):	5.00	Humidity (%): 50
Crosshead Speed (mm/min):	10.000	Temperature (deg. C): -29

Sample Description: 2388

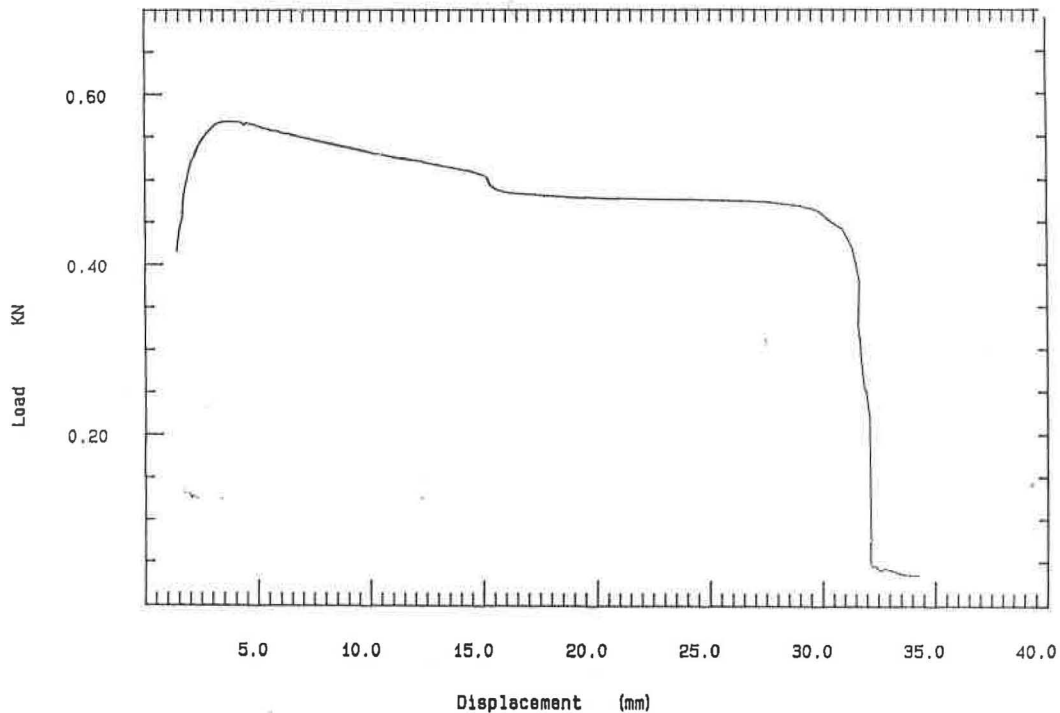
Dimensions:

	<u>Spec. 1</u>	<u>Spec. 2</u>	<u>Spec.3</u>	<u>Spec. 4</u>
Diameter (mm)	25.400	25.400	25.400	25.400
Spec gauge len (mm)	25.797	25.438	25.439	25.756
Grip distance (mm)	125.000	125.000	125.000	125.000

Out of 4 specimens, 1 excluded.

Specimen No.	Toughness (J)	Peak Load (N)	Ratio of Toughness	Displacement at Max. Load (mm)	Maximum Displacement (mm)
1	15.090	627.4	.0240	3.503	30.33
2	19.480	576.4	.0338	5.152	38.72
Excluded	6.719	594.1	.0113	3.681	14.55
4	15.530	568.6	.0273	3.434	33.87
Mean	16.700	590.8	.0284	4.030	34.31
Std. Deviation	2.415	31.9	.0050	.973	4.21

FIGURE 28. Tensile Adhesion at -30C, Sample 2388



Adhesion Test Using T&T System - TAC Proposal for C&J Test

Test Type:	Tensile	Instron Corporation
Sample Identification:	AT2389A1	Series IX Automated Materials Testing System 1.02C
Interface Type:	4200 Series	Test Date: 23 Sep 1994
Machine Parameters of Test:		Sample Type: ASTM
Sample rate (pts/sec):	5.00	Humidity (%): 50
Crosshead Speed (mm/min):	10.000	Temperature (deg. C): -29

Sample Description: 2389

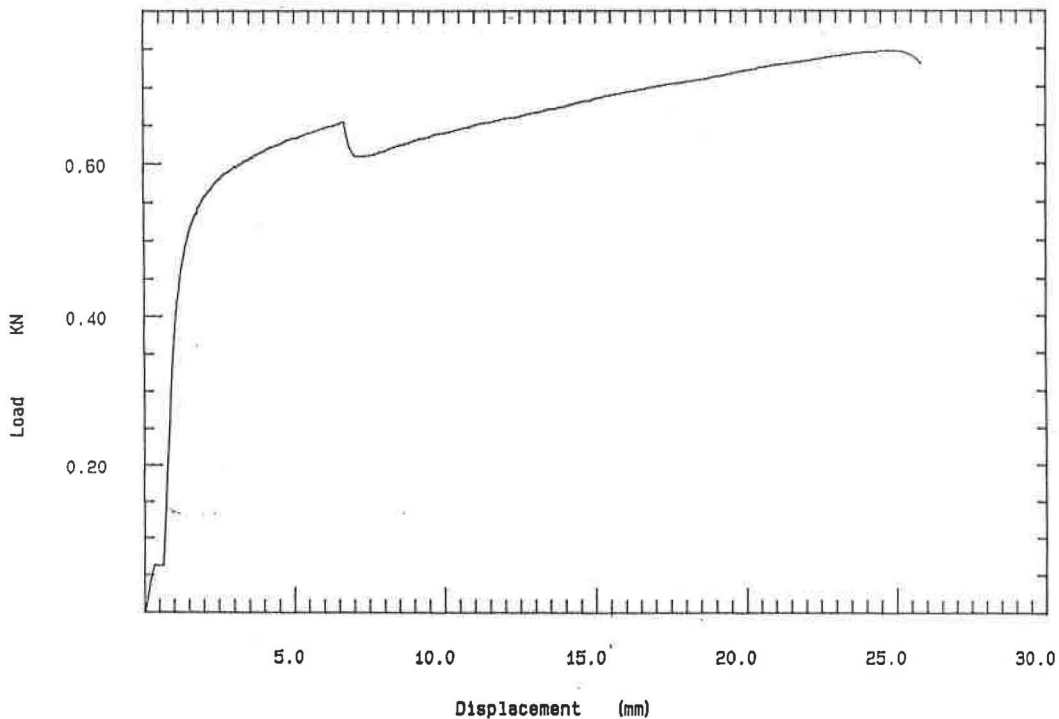
Dimensions:

	<u>Spec. 1</u>	<u>Spec. 2</u>	<u>Spec.3</u>	<u>Spec. 4</u>
Diameter (mm)	25.400	25.400	25.400	25.400
Spec gauge len (mm)	25.961	26.279	26.453	26.028
Grip distance (mm)	125.000	125.000	125.000	125.000

Out of 4 specimens, 1 excluded.

Specimen No.	Toughness (J)	Peak Load (N)	Ratio of Toughness	Displacement at Max. Load (mm)	Maximum Displacement (mm)
1	16.58	751.4	.0221	24.37	25.26
2	14.10	713.3	.0198	18.01	22.18
Excluded	10.55	714.9	.0148	14.16	16.81
4	16.08	727.0	.0221	23.54	25.80
Mean	15.58	730.6	.0213	21.97	24.41
Std. Deviation	1.31	19.3	.0013	3.46	1.95

FIGURE 29. Tensile Adhesion at -30C, Sample 2389



Adhesion Test Using T&T System - TAC Proposal for C&J Test

Test Type:	Tensile	Instron Corporation
Sample Identification:		Series IX Automated Materials Testing System 1.02C
Interface Type:	4200 Series	Test Date: 23 Sep 1994
Machine Parameters of Test:		Sample Type: ASTM
Sample rate (pts/sec):	5.00	Humidity (%): 50
Crosshead Speed (mm/min):	10.000	Temperature (deg. C): -29

Sample Description: 2390

Dimensions:

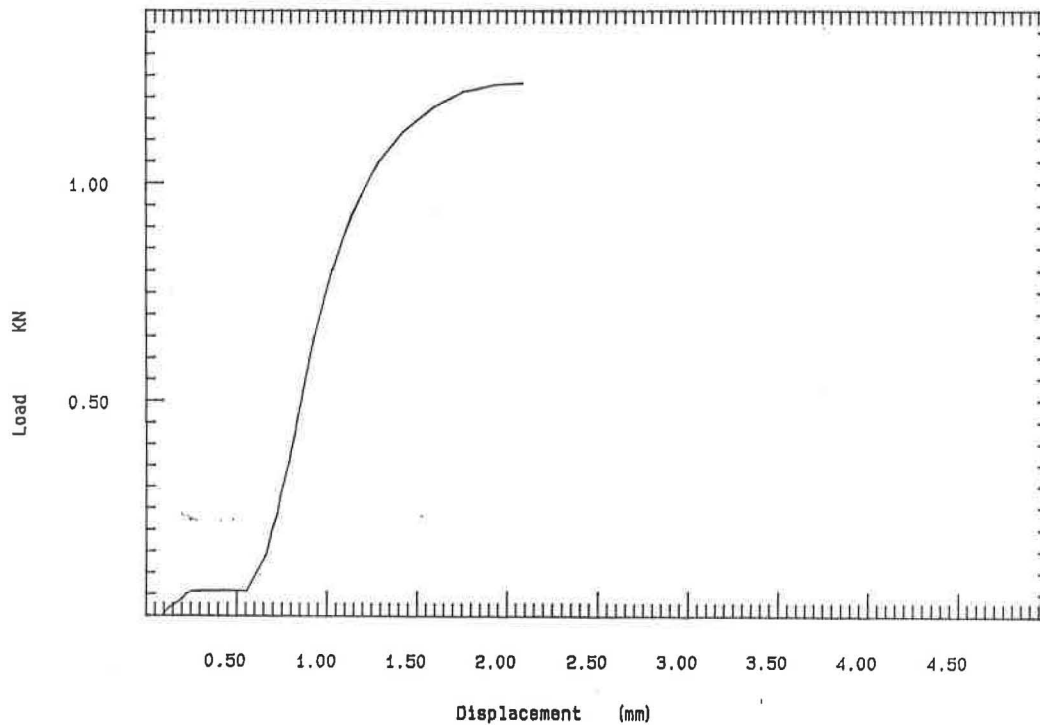
	Spec. 1	Spec. 2	Spec.3	Spec. 4
Diameter (mm)	25.400	25.400	25.400	25.400
Spec gauge len (mm)	25.993	26.022	26.898	26.098
Grip distance (mm)	125.000	125.000	125.000	125.000

Out of 4 specimens, 0 excluded.

Sample Comments: Sample broke, did NOT detach from brick

Specimen No.	Toughness (J)	Peak Load (N)	Ratio of Toughness	Displacement at Max. Load (mm)	Maximum Displacement (mm)
1	.5476	1023.	.0005	.9267	.8020
2	1.3940	1238.	.0011	1.4380	1.4680
3	2.9650	1111.	.0027	2.9520	3.1940
4	1.4690	1207.	.0012	1.2120	1.7590
Mean	1.5940	1145.	.0014	1.6320	1.8060
Std. Deviation	1.0050	98.	.0009	.9044	1.0080

FIGURE 30. Tensile Adhesion at -30C, Sample 2390



Adhesion Test Using T&T System - TAC Proposal for C&J Test

Test Type:	Tensile	Instron Corporation
Sample Identification:	879248P04	Series IX Automated Materials Testing System 1.02C
Interface Type:	4200 Series	Test Date: 30 Sep 1994
Machine Parameters of Test:		Sample Type: ASTM
Sample rate (pts/sec):	5.00	Humidity (%): 50
Crosshead Speed (mm/min):	10.000	Temperature (deg. C): -29

Sample Description: 2448

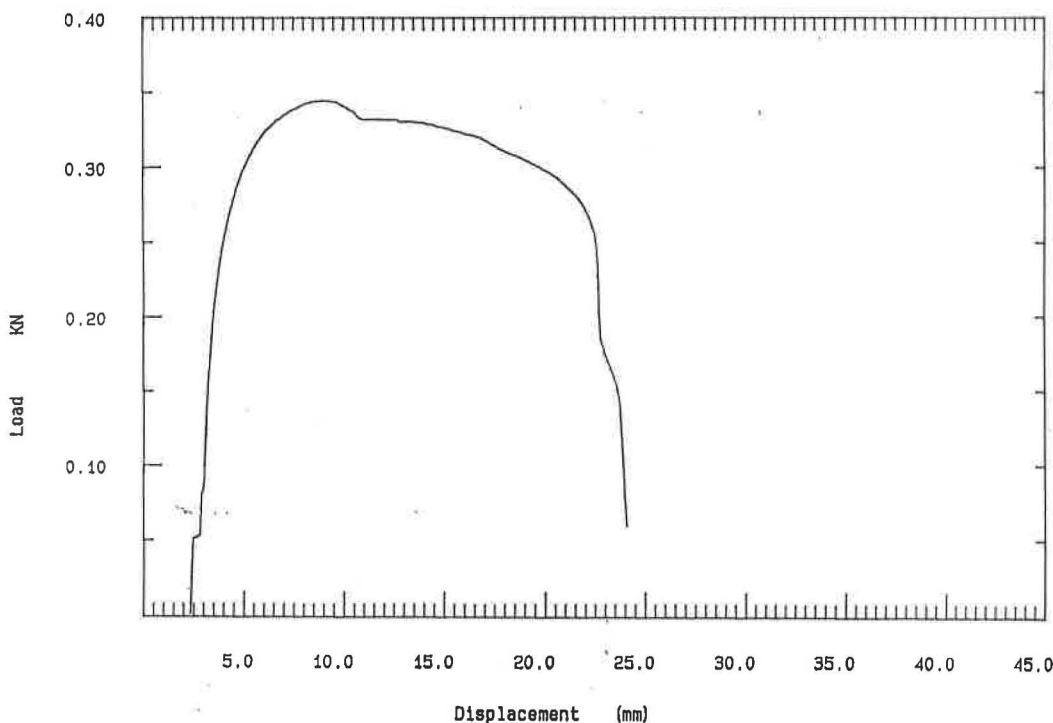
Dimensions:

	Spec. 1	Spec. 2	Spec.3	Spec. 4	Spec. 5	Spec. 6
Diameter (mm)	25.400	25.400	25.400	25.400	25.400	25.400
Spec gauge len (mm)	25.418	26.517	27.399	27.687	25.642	25.751
Grip distance (mm)	125.000	125.000	125.000	125.000	125.000	125.000

Out of 6 specimens, 2 excluded.

Specimen No.	Toughness (J)	Peak Load (N)	Ratio of Toughness	Displacement at Max. Load (mm)	Maximum Displacement (mm)
1	6.3530	345.90	.0184	7.8520	23.3400
Excluded	-.0011	40.13	.0000	.3026	.2726
Excluded	.0034	50.06	.0001	.2505	.2385
4	6.3500	345.80	.0184	7.1430	21.7700
5	11.1500	304.00	.0367	13.6700	41.1900
6	5.2760	296.40	.0178	5.0890	23.9400
Mean	7.2810	232.00	.0228	8.4380	27.5600
Std. Deviation	2.6270	26.54	.0093	3.6780	9.1320

FIGURE 31. Tensile Adhesion at -30C, Sample 2448



Adhesion Test Using T&T System - TAC Proposal for C&J Test

Test Type:	Tensile	Instron Corporation
Sample Identification:	AT2449R1	Series IX Automated Materials Testing System 1.02C
Interface Type:	4200 Series	Test Date: 30 Sep 1994
Machine Parameters of Test:		Sample Type: ASTM
Sample rate (pts/sec):	5.00	Humidity (%): 50
Crosshead Speed (mm/min):	10.000	Temperature (deg. C): -29

Sample Description: 2449

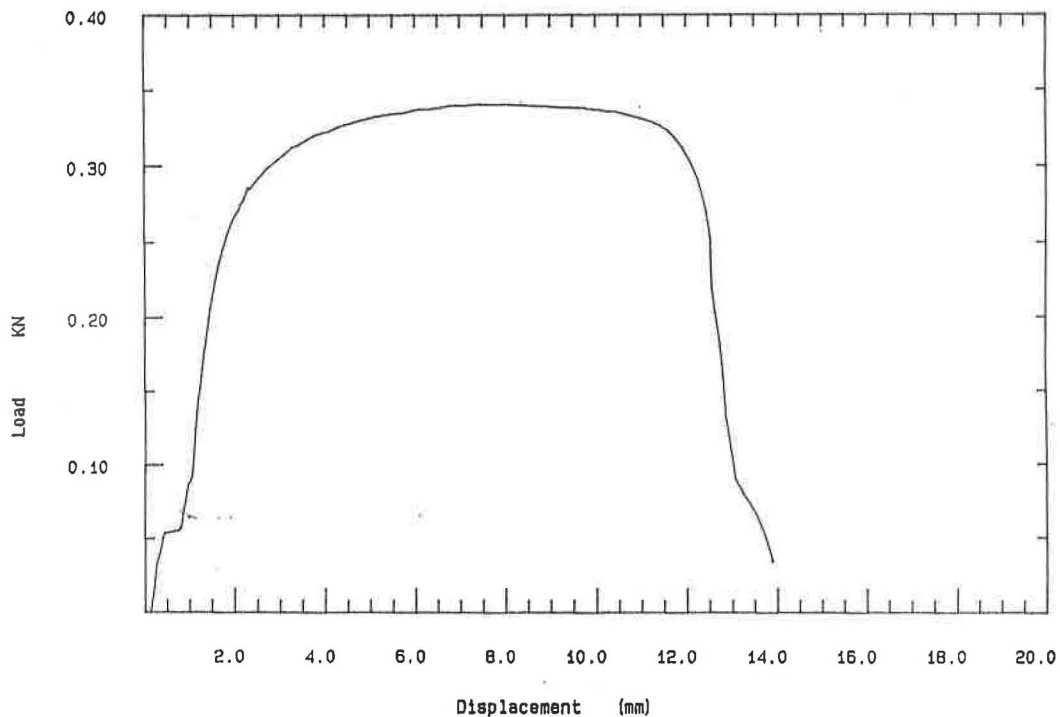
Dimensions:

	<u>Spec. 1</u>	<u>Spec. 2</u>
Diameter (mm)	25.400	25.400
Spec gauge len (mm)	25.521	25.502
Grip distance (mm)	125.000	125.000

Out of 2 specimens, 0 excluded.

Specimen No.	Toughness (J)	Peak Load (N)	Ratio of Toughness	Displacement at Max. Load (mm)	Maximum Displacement (mm)
1	3.804	343.4	.0111	6.829	13.77
2	4.964	349.4	.0142	6.728	17.46
Mean	4.384	346.4	.0126	6.779	15.62
Std. Deviation	.820	4.2	.0022	.072	2.61

FIGURE 32. Tensile Adhesion at -30C, Sample 2449



Adhesion Test Using T&T System - TAC Proposal for C&J Test

Test Type:	Tensile	Instron Corporation
Sample Identification:		Series IX Automated Materials Testing System 1.02C
Interface Type:	4200 Series	Test Date: 03 Oct 1994
Machine Parameters of Test:		Sample Type: ASTM
Sample rate (pts/sec):	5.00	Humidity (%): 50
Crosshead Speed (mm/min):	10.000	Temperature (deg. C): -29

Sample Description: 2450

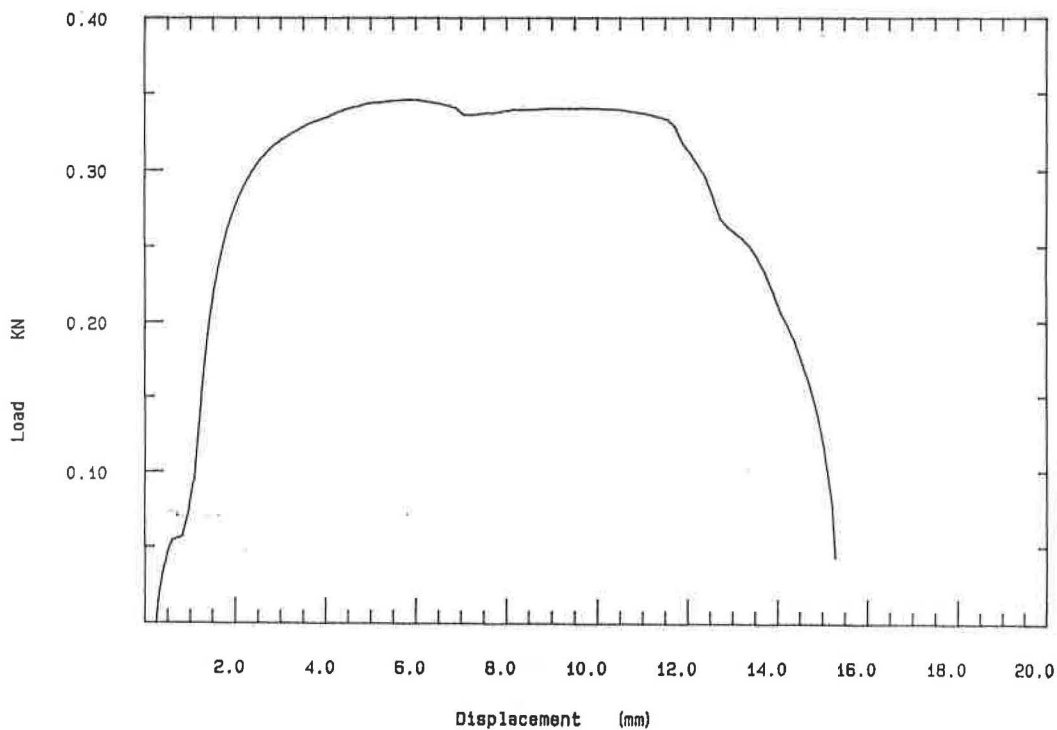
Dimensions:

	<u>Spec. 1</u>	<u>Spec. 2</u>	<u>Spec. 3</u>	<u>Spec. 4</u>
Diameter (mm)	25.400	25.400	25.400	25.400
Spec gauge len (mm)	25.379	25.520	26.195	25.522
Grip distance (mm)	125.000	125.000	125.000	125.000

Out of 4 specimens, 0 excluded.

Specimen No.	Toughness (J)	Peak Load (N)	Ratio of Toughness	Displacement at Max. Load (mm)	Maximum Displacement (mm)
1	3.093	367.5	.0084	5.301	11.340
2	5.821	366.6	.0159	8.330	18.640
3	4.303	346.2	.0124	5.125	14.490
4	2.253	379.6	.0059	3.878	9.467
Mean	3.868	365.0	.0107	5.658	13.490
Std. Deviation	1.550	13.8	.0044	1.891	4.015

FIGURE 33. Tensile Adhesion at -30C, Sample 2450



Adhesion Test Using T&T System - TAC Proposal for C&J Test

Test Type:	Tensile	Instron Corporation
Sample Identification:		Series IX Automated Materials Testing System 1.02C
Interface Type:	4200 Series	Test Date: 04 Oct 1994
Machine Parameters of Test:		Sample Type: ASTM
Sample rate (pts/sec):	5.00	Humidity (%): 50
Crosshead Speed (mm/min):	10.000	Temperature (deg. C): -29

Sample Description: 2451

Dimensions:

	<u>Spec. 1</u>	<u>Spec. 2</u>	<u>Spec.3</u>	<u>Spec. 4</u>
Diameter (mm)	25.400	25.400	25.400	25.400
Spec gauge len (mm)	25.636	25.605	25.677	25.564
Grip distance (mm)	125.000	125.000	125.000	125.000

Out of 4 specimens, 0 excluded.

Specimen No.	Toughness (J)	Peak Load (N)	Ratio of Toughness	Displacement at Max. Load (mm)	Maximum Displacement (mm)
1	4.056	272.9	.0149	9.404	17.82
2	5.305	316.9	.0167	12.820	19.26
3	13.360	362.8	.0368	10.070	41.72
4	9.457	365.1	.0259	8.986	29.53
Mean	8.045	329.4	.0236	10.320	27.08
Std. Deviation	4.230	43.7	.0101	1.728	11.06

FIGURE 34. Tensile Adhesion at -30C, Sample 2451

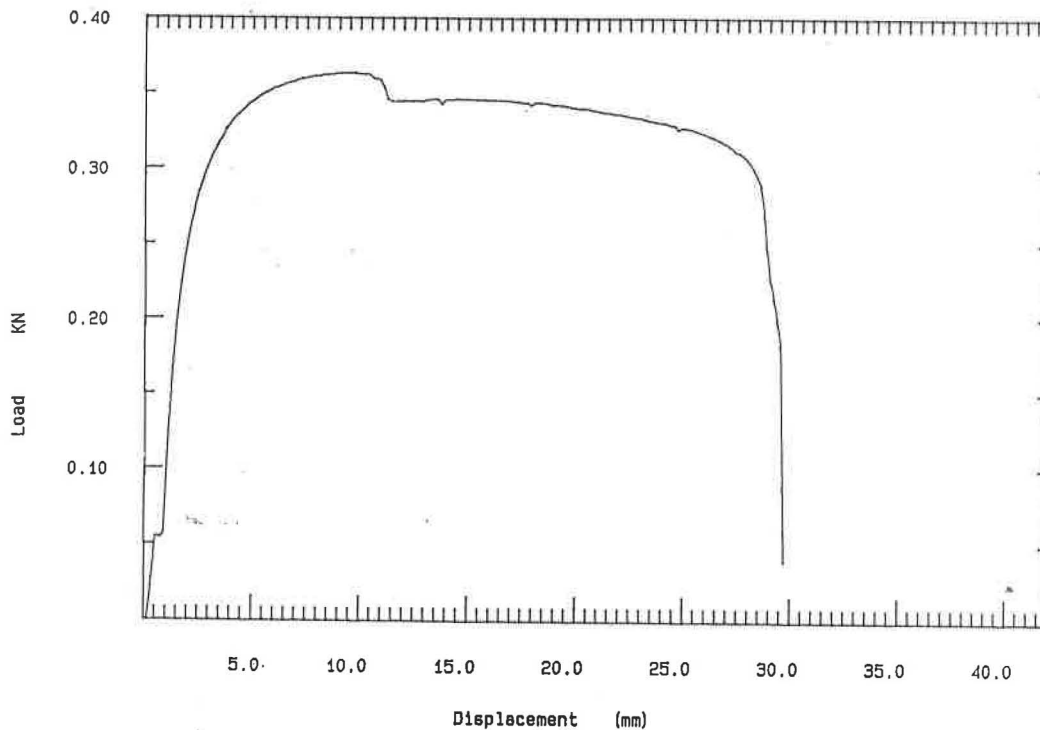


FIGURE 35. Hot Oil Heater for Crack Sealants

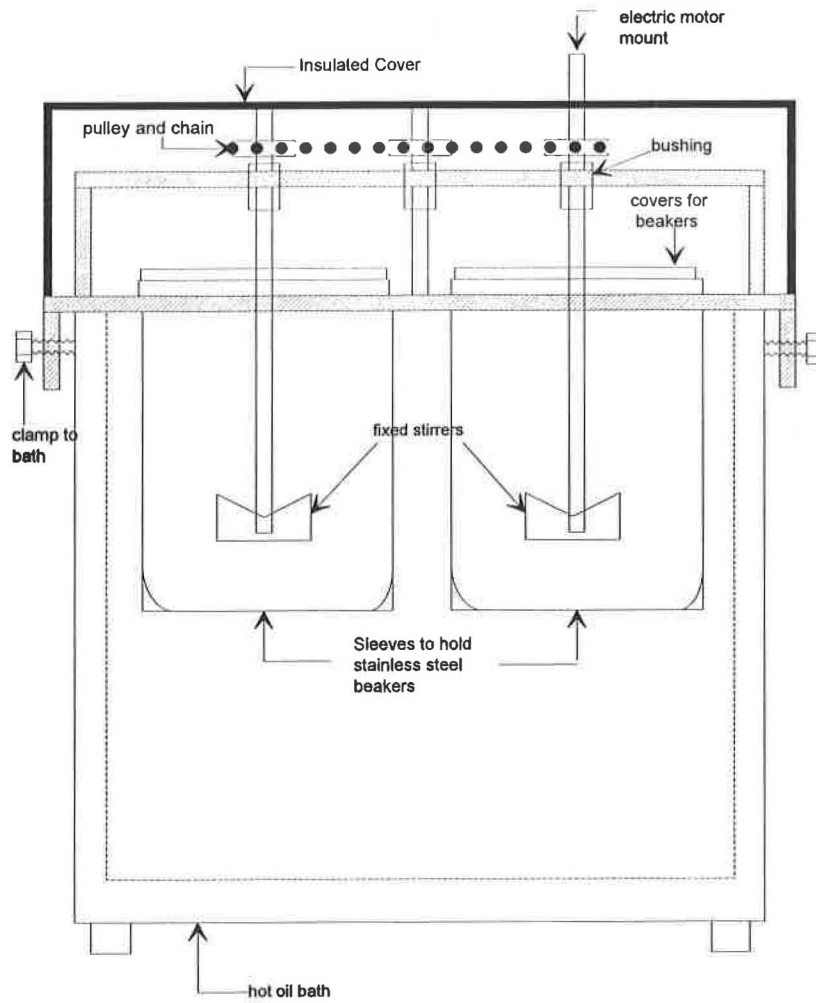


FIGURE 36. Stress Relaxation at -30°C, Sample 1

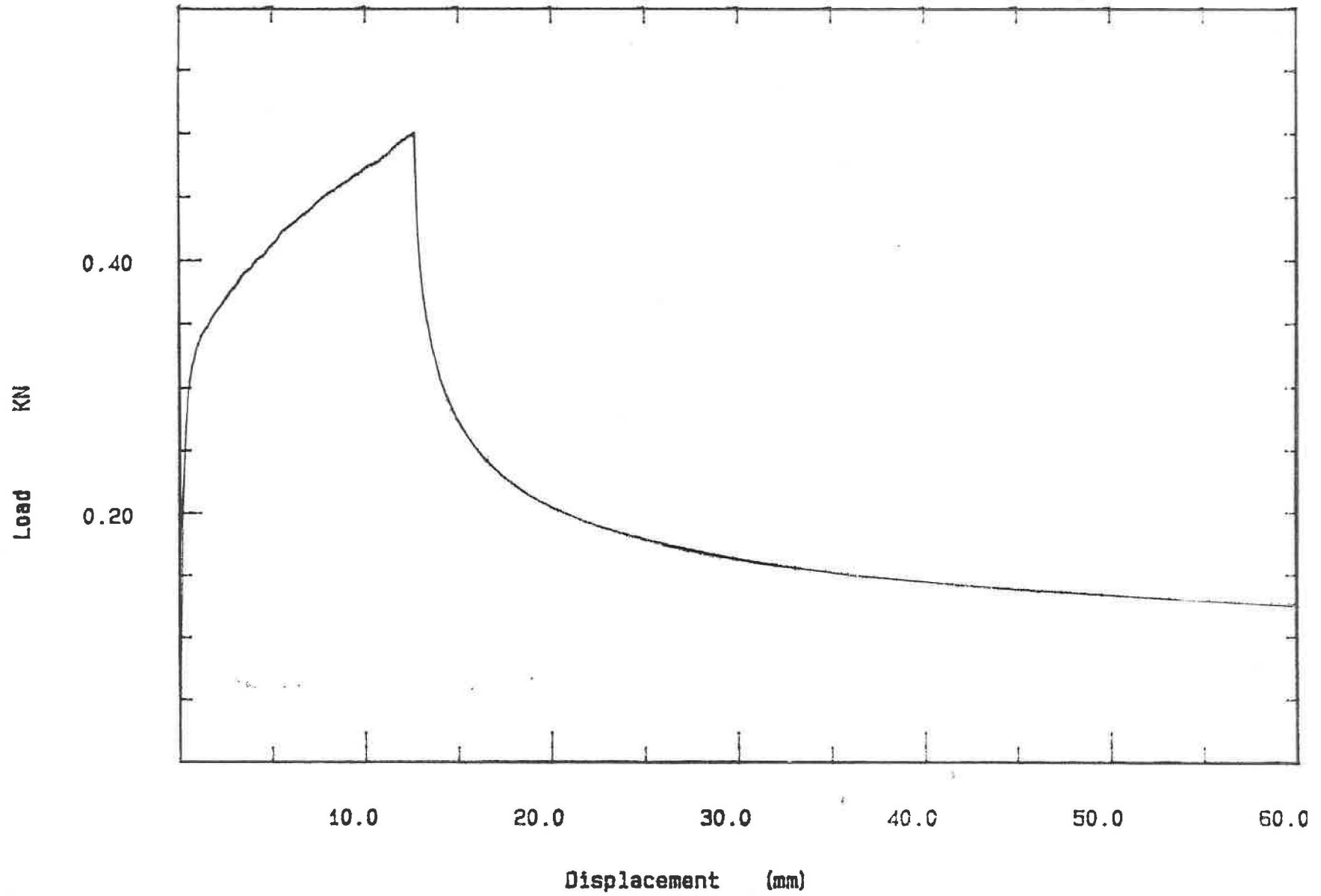


FIGURE 37. Stress Relaxation at -30°C, Sample 2

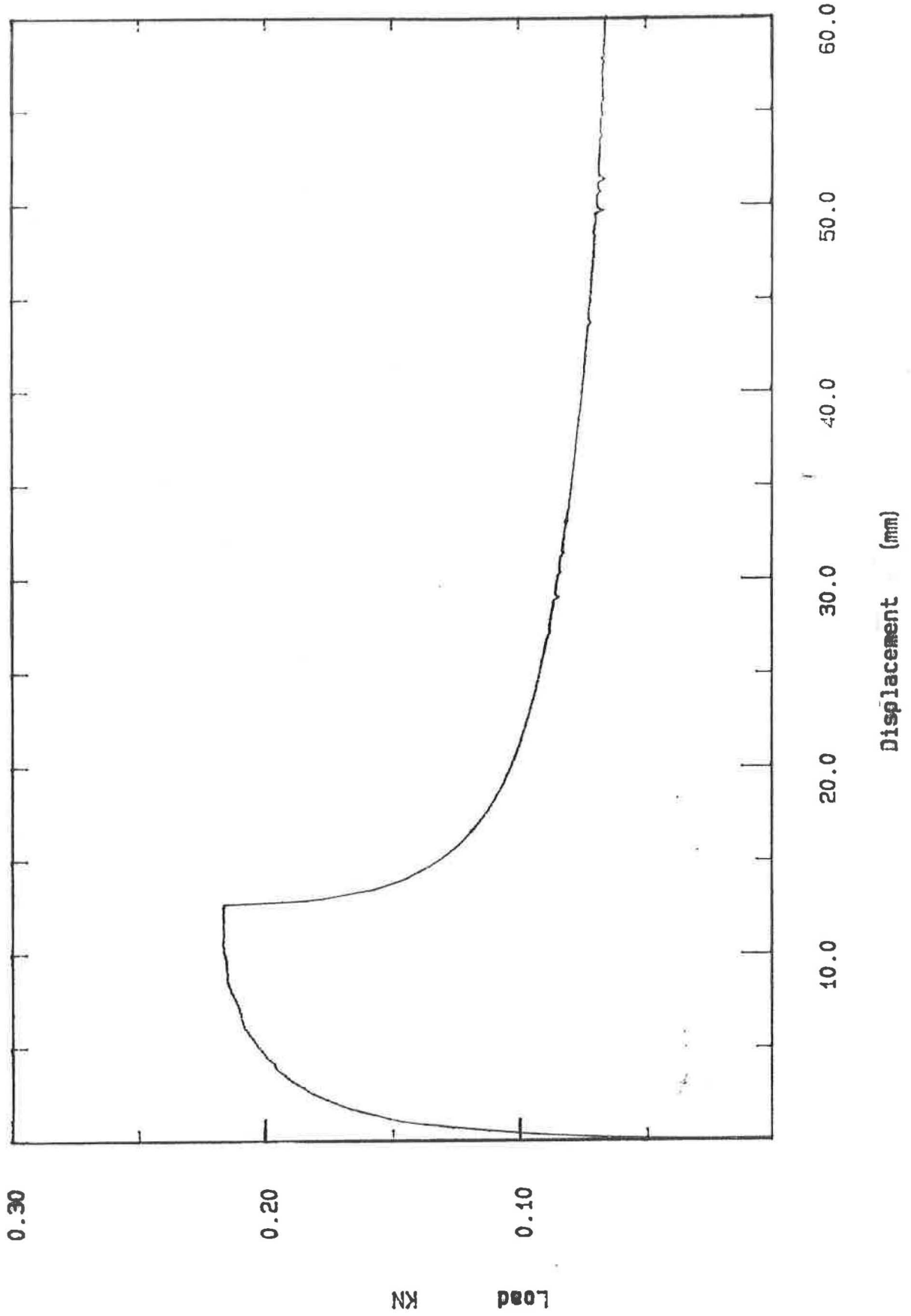


FIGURE 38. Stress Relaxation at -30°C, Sample 3

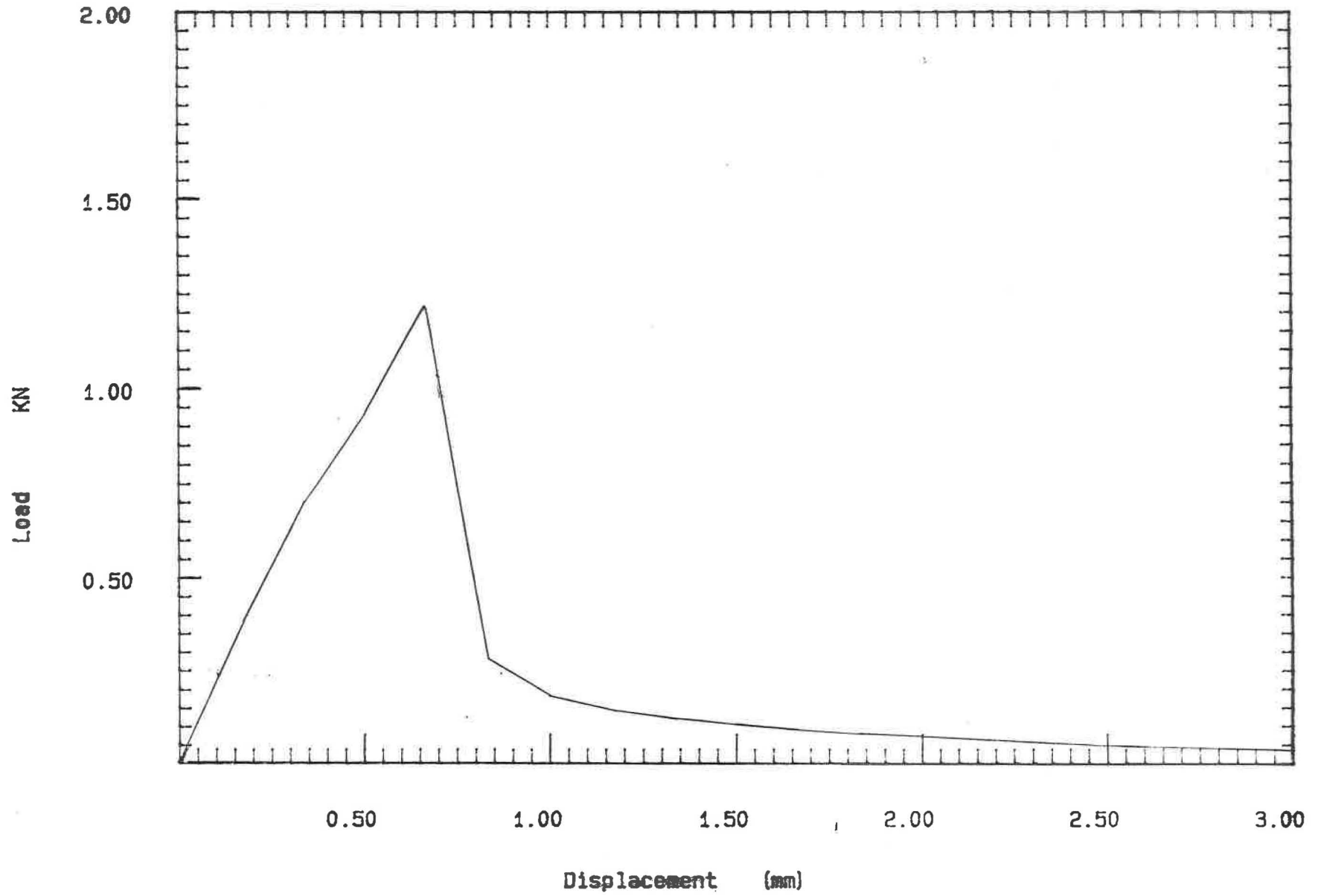


FIGURE 39. Stress Relaxation at -30°C, Sample 4

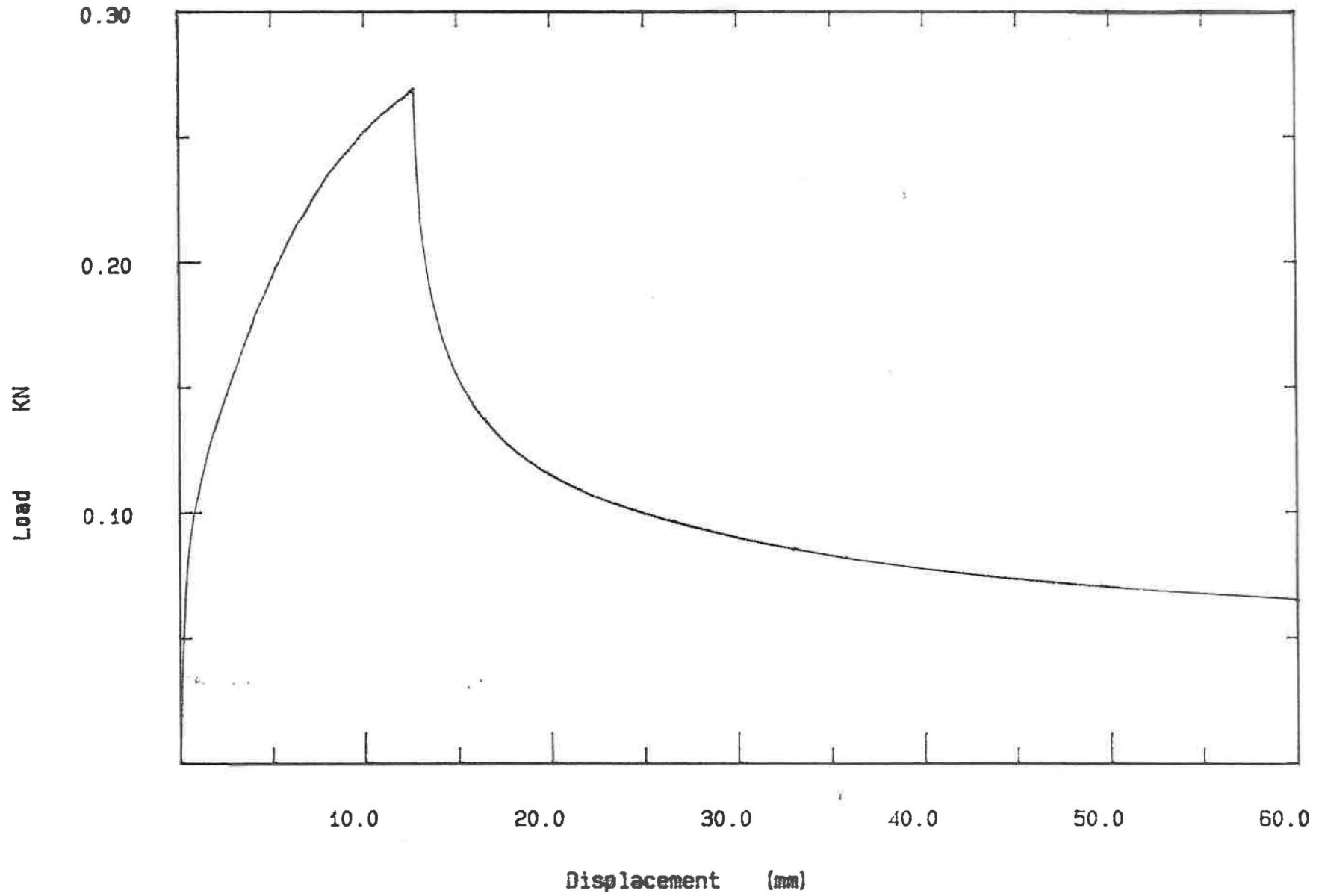


FIGURE 40. Stress Relaxation at -30°C, Sample 5

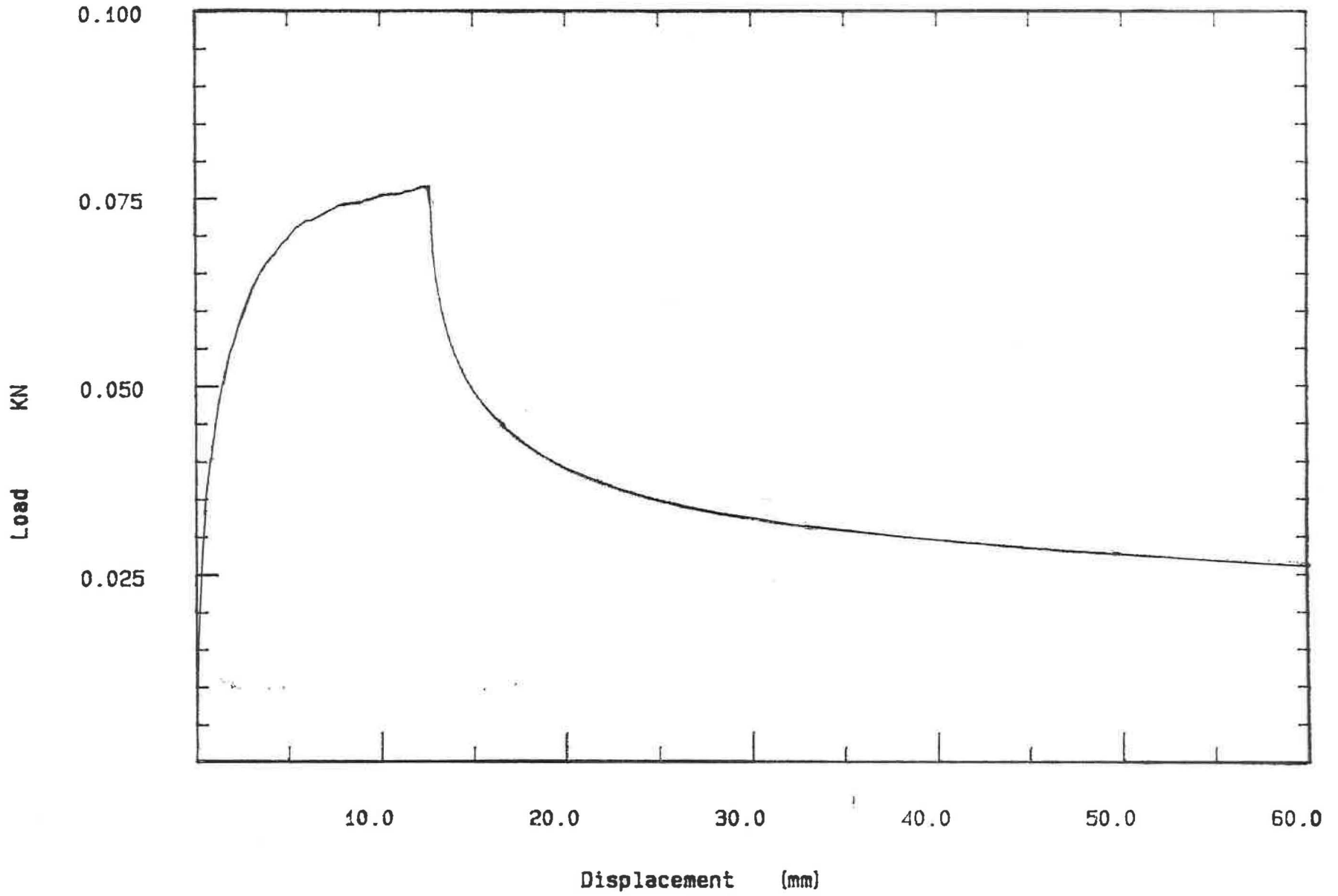


FIGURE 41. Stress Relaxation at -30°C, Sample 6

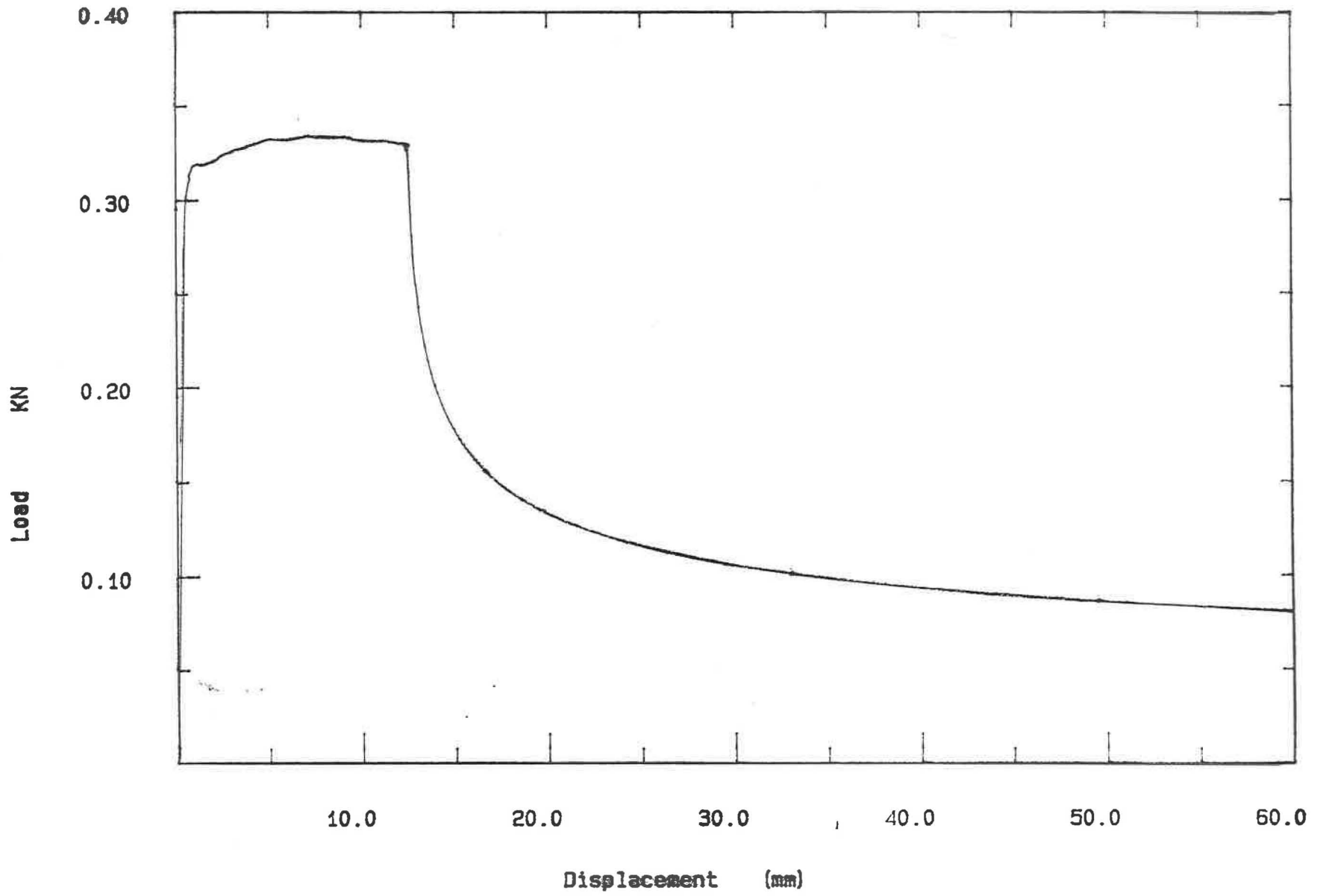


FIGURE 42. Stress Relaxation at -30°C, Sample 7

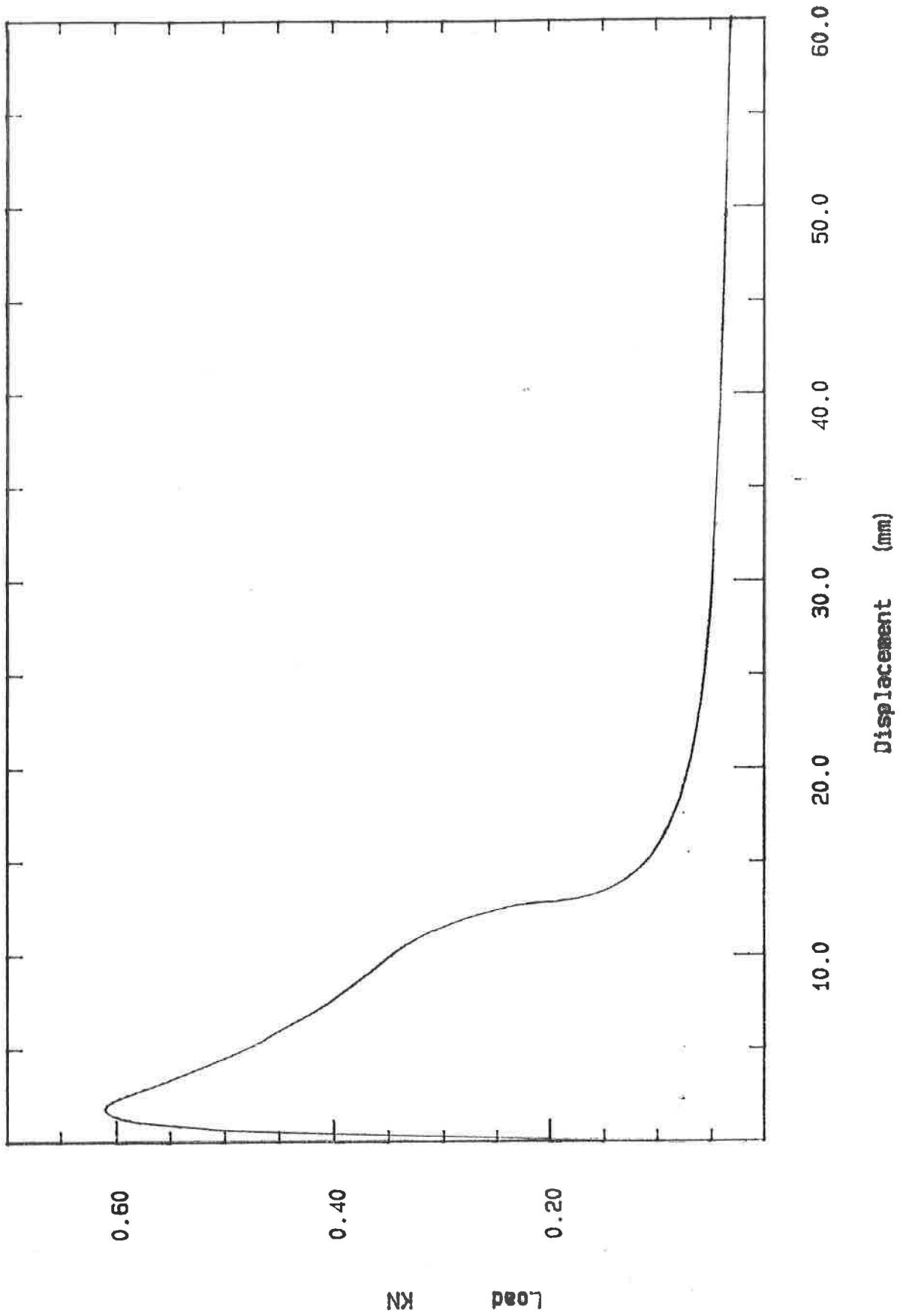


FIGURE 43. Stress Relaxation at -30°C, Sample 8

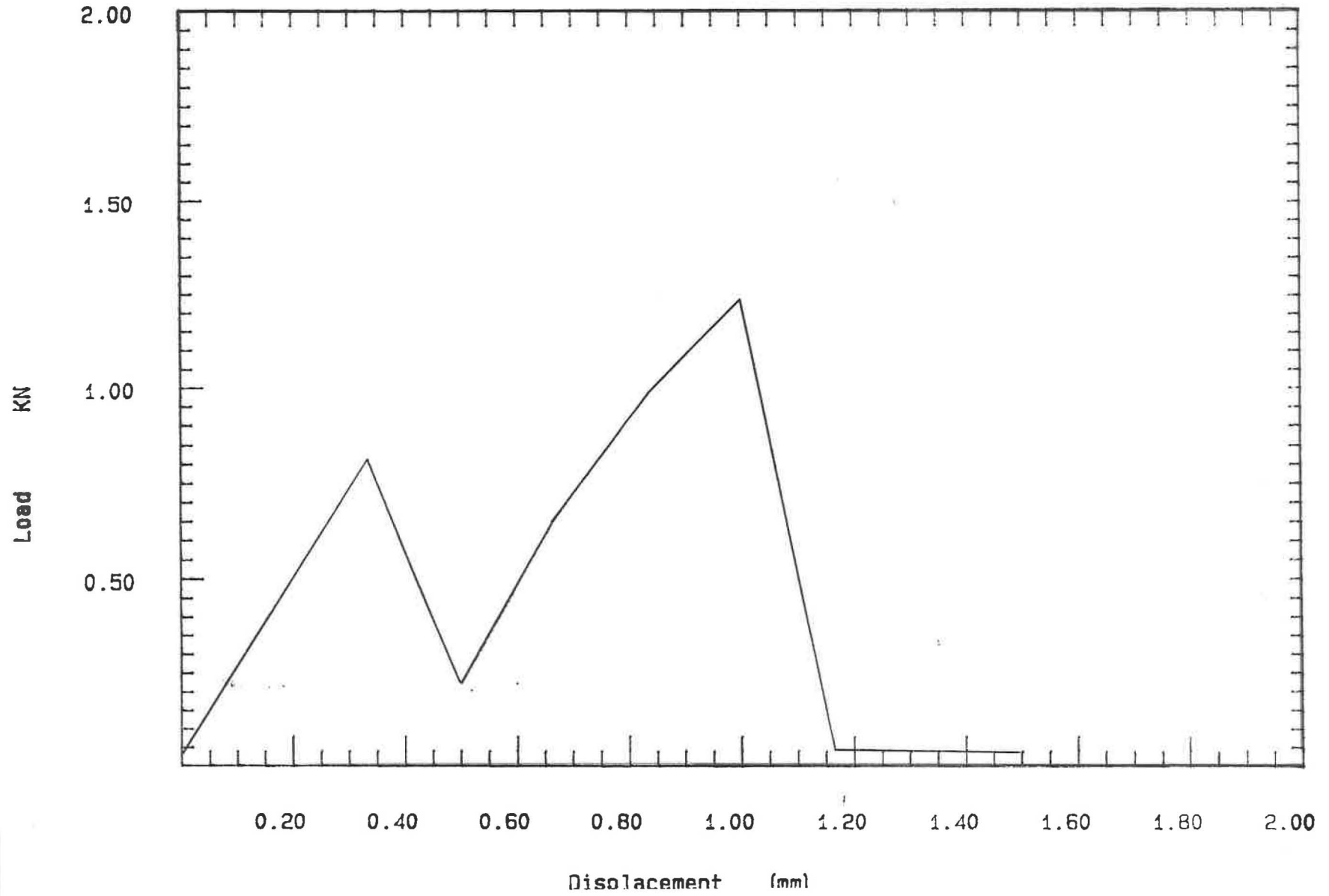


Figure 44. Stress Relaxation at -30°C, Sample 9

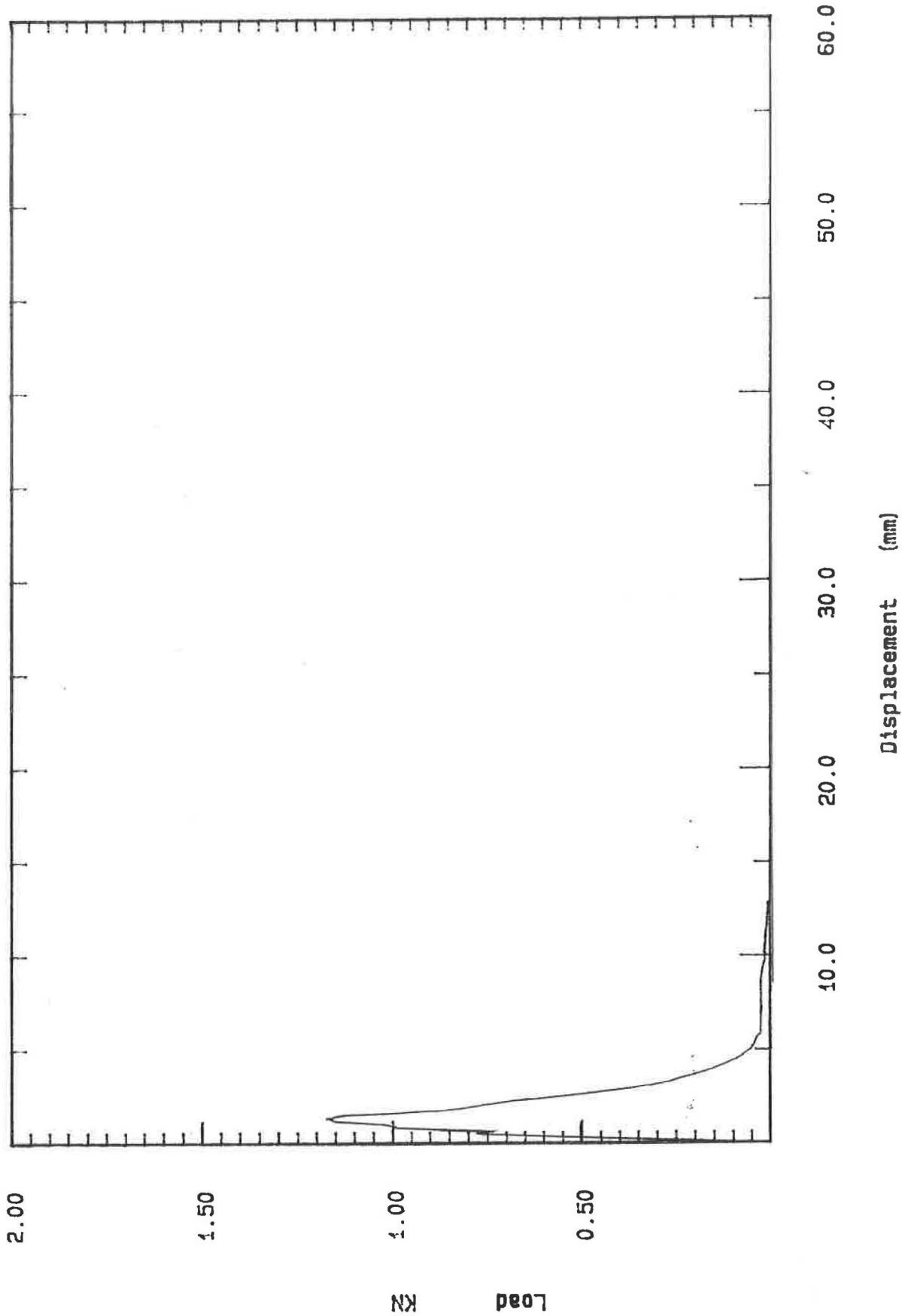


FIGURE 45. Stress Relaxation at -30°C, Sample 10

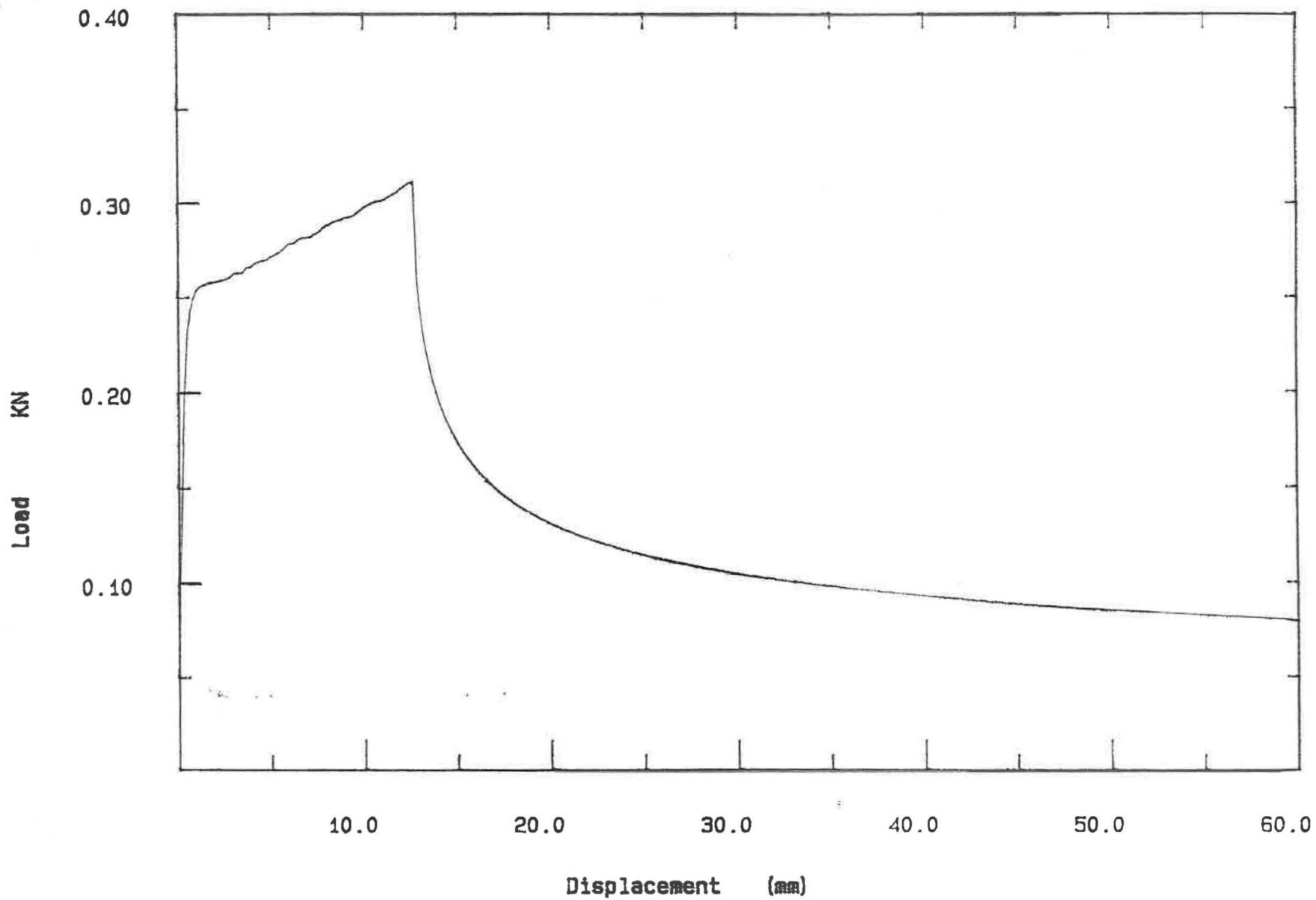


FIGURE 46. Stress Relaxation at -30°C, Sample 11

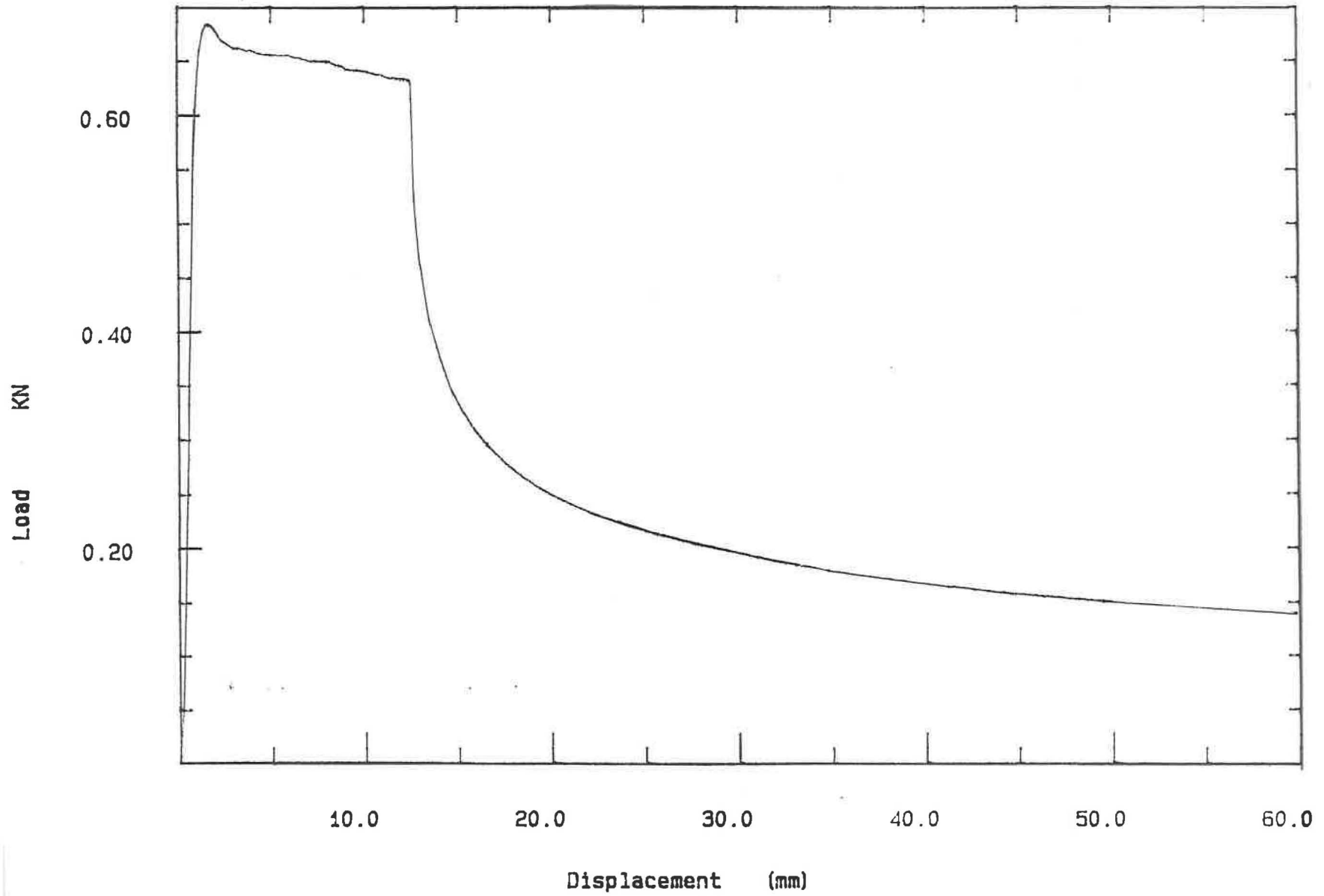


FIGURE 47. Stress Relaxation at -30°C, Sample 12

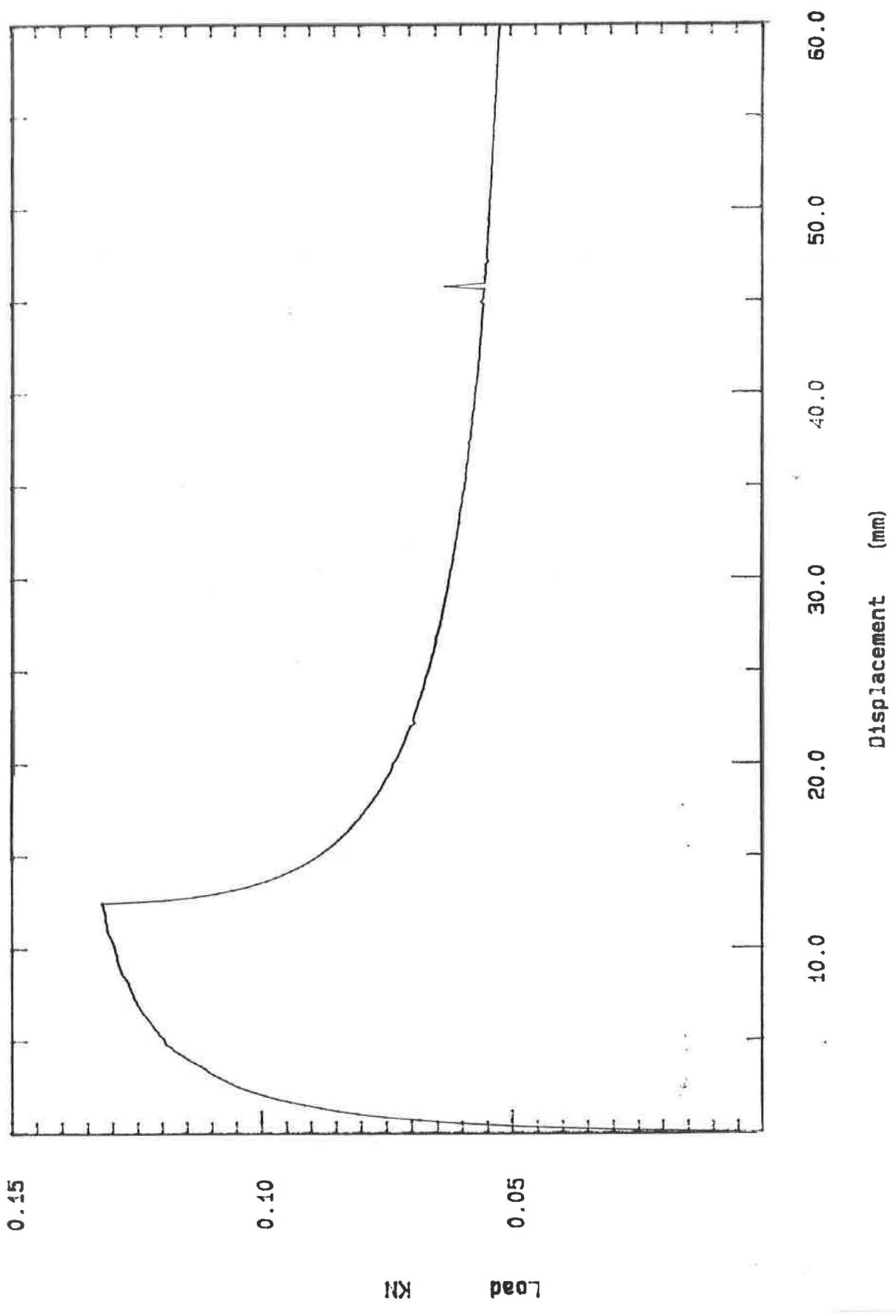


FIGURE 48. Stress Relaxation at -30°C, Sample 13

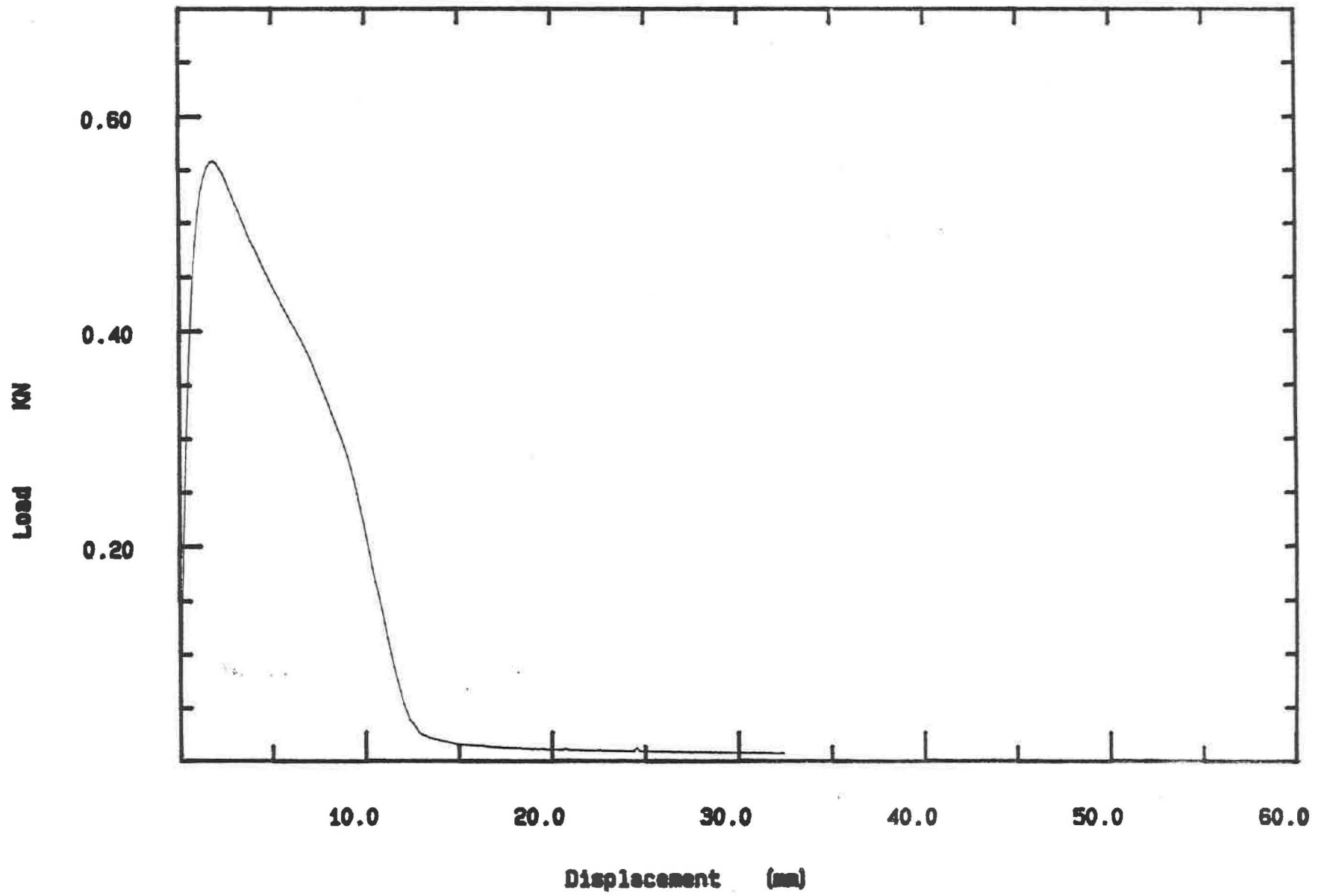
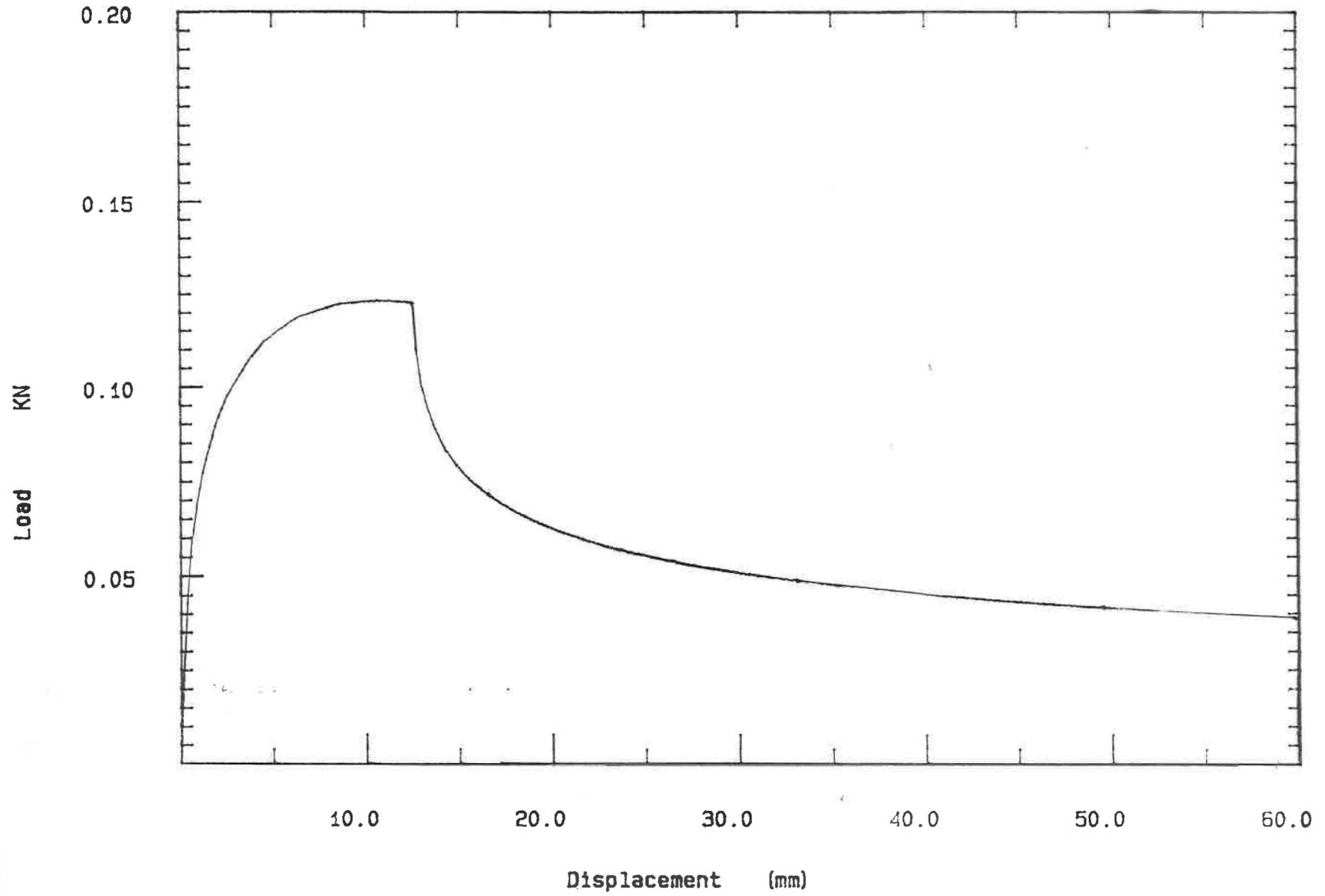


FIGURE 49. Stress Relaxation at -30°C, Sample 14



Adhesion Test Using T&T System - TAC Proposal for C&J Test

Test Type:	Tensile	Instron Corporation
Sample Identification:		Series IX Automated Materials Testing System 1.02C
Interface Type:	4200 Series	Test Date: 16 Dec 1995
Machine Parameters of Test:		Sample Type: ASTM
Sample rate (pts/sec):	5.00	Humidity (%): 50
Crosshead Speed (mm/min):	10.000	Temperature (deg. C): -30

Sample Description: 1

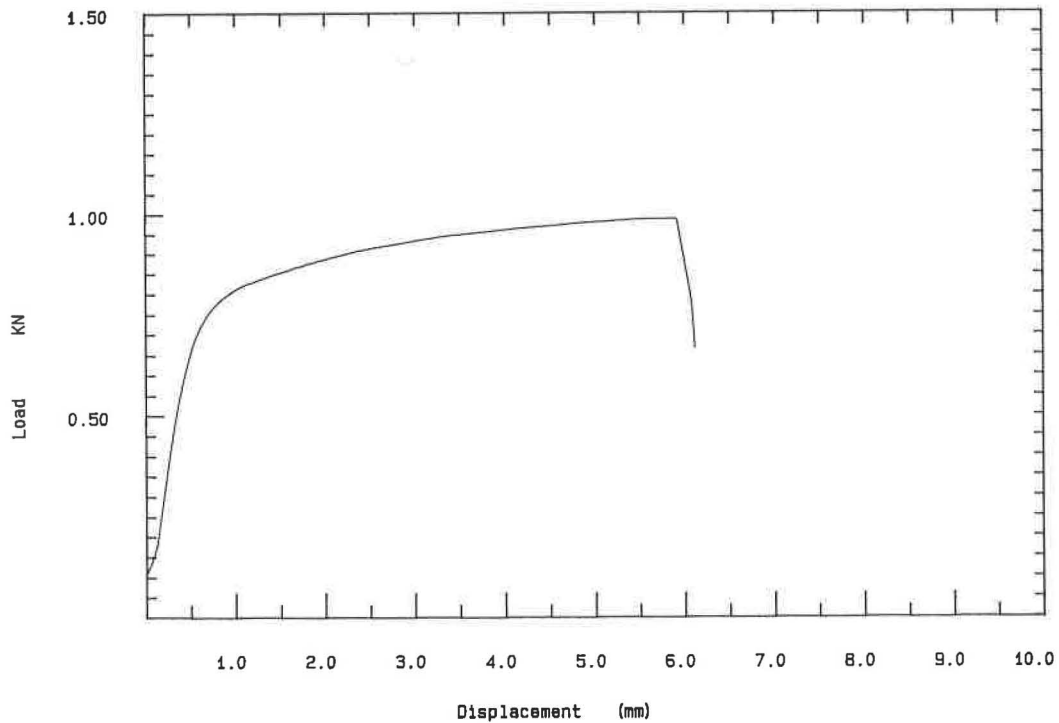
Dimensions:

	Spec. 1	Spec. 2	Spec.3	Spec. 4	Spec. 5
Diameter (mm)	25.400	25.400	25.400	25.400	25.400
Spec gauge len (mm)	26.229	25.777	25.400	26.210	27.326
Grip distance (mm)	125.000	125.000	125.000	125.000	125.000

Out of 5 specimens, 2 excluded.

Specimen No.	Toughness (J)	Peak Load (N)	Ratio of Toughness	Displacement at Max. Load (mm)	Maximum Displacement (mm)
Excluded	6.3590	1526.00	.0042	3.231	6.025
2	4.7690	979.20	.0049	5.433	6.118
Excluded	.0030	13.82	.0002	1.140	1.124
4	8.6900	1043.00	.0083	6.140	9.313
5	16.2600	976.50	.0167	1.434	17.630
Mean	9.9070	999.60	.0100	4.336	11.020
Std. Deviation	5.8420	37.64	.0061	2.538	5.944

FIGURE 60. Tensile Adhesion at -30°C, Sample 1



Adhesion Test Using T&T System - TAC Proposal for C&J Test

Test Type:	Tensile	Instron Corporation
Sample Identification:		Series IX Automated Materials Testing System 1.02C
Interface Type:	4200 Series	Test Date: 09 Dec 1995
Machine Parameters of Test:		Sample Type: ASTM
Sample rate (pts/sec):	5.00	Humidity (%): 50
Crosshead Speed (mm/min):	10.000	Temperature (deg. C): -30

Sample Description: 2

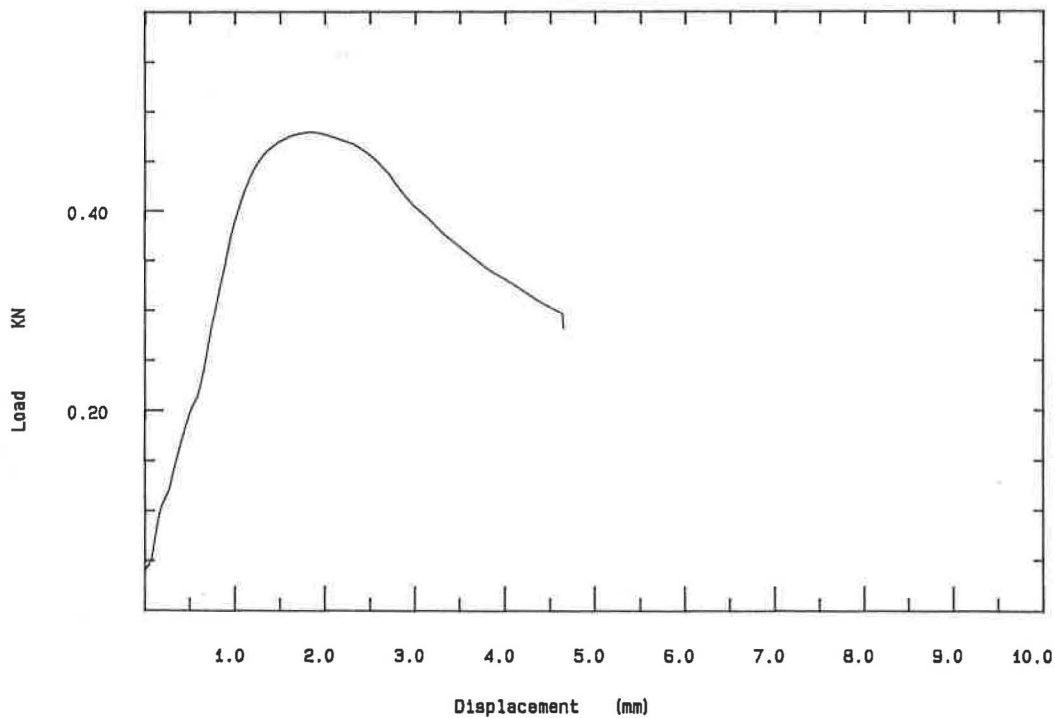
Dimensions:

	Spec. 1	Spec. 2	Spec.3	Spec. 4
Diameter (mm)	25.400	25.400	25.400	25.400
Spec gauge len (mm)	25.440	28.203	25.818	28.152
Grip distance (mm)	125.000	125.000	125.000	125.000

Out of 4 specimens, 0 excluded.

Specimen No.	Toughness (J)	Peak Load (N)	Ratio of Toughness	Displacement at Max. Load (mm)	Maximum Displacement (mm)
1	1.167	478.1	.0024	1.670	4.654
2	1.775	451.1	.0039	1.847	6.359
3	3.660	520.3	.0070	2.722	9.076
4	3.084	487.2	.0063	2.318	7.507
Mean	2.421	484.2	.0049	2.139	6.899
Std. Deviation	1.150	28.5	.0021	.475	1.866

FIGURE 51. Tensile Adhesion at -30°C, Sample 2



Adhesion Test Using T&T System - TAC Proposal for C&J Test

Test Type:	Tensile	Instron Corporation
Sample Identification:		Series IX Automated Materials Testing System 1.02C
Interface Type:	4200 Series	Test Date: 09 Dec 1995
Machine Parameters of Test:		Sample Type: ASTM
Sample rate (pts/sec):	5.00	Humidity (%): 50
Crosshead Speed (mm/min):	10.000	Temperature (deg. C): -30

Sample Description: 3

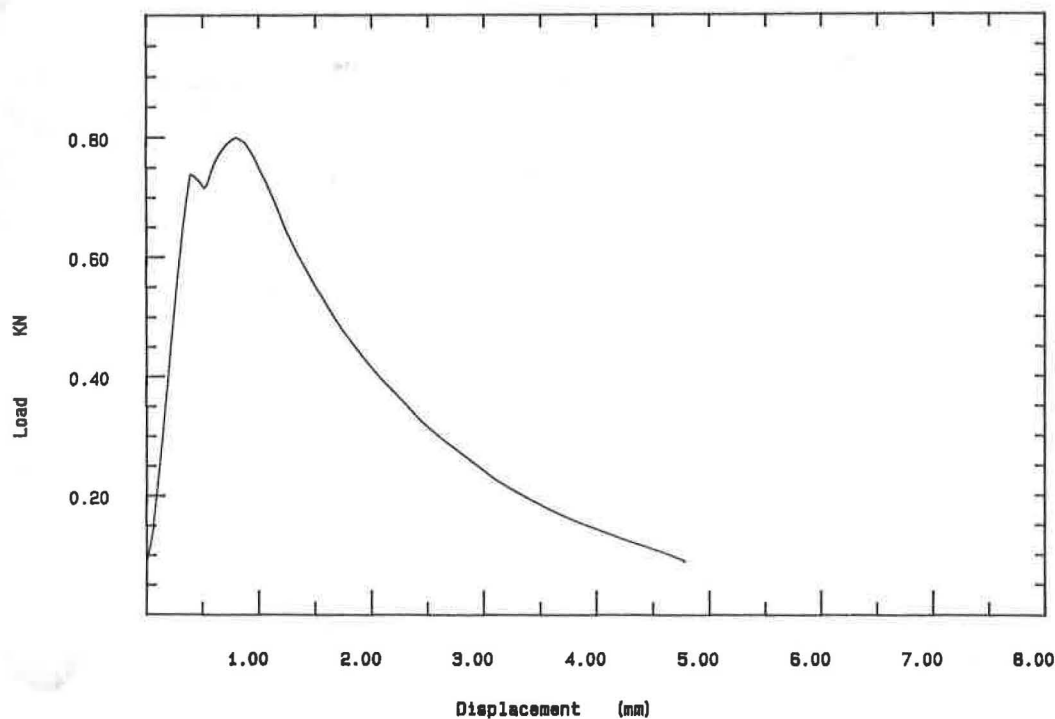
Dimensions:

	<u>Spec. 1</u>	<u>Spec. 2</u>	<u>Spec.3</u>	<u>Spec. 4</u>
Diameter (mm)	25.400	25.400	25.400	25.400
Spec gauge len (mm)	26.137	25.897	27.838	26.459
Grip distance (mm)	125.000	125.000	125.000	125.000

Out of 4 specimens, 1 excluded.

Specimen No.	Toughness (J)	Peak Load (N)	Ratio of Toughness	Displacement at Max. Load (mm)	Maximum Displacement (mm)
1	3.0530	1057.0	.0029	.9333	6.9530
2	.3500	1120.0	.0003	.5433	.4940
Excluded	.5697	745.5	.0008	.4319	4.7830
4	2.0640	1070.0	.0019	.7710	7.5680
Mean	1.8220	1082.0	.0017	.7492	5.0050
Std. Deviation	1.3680	33.3	.0013	.1959	3.9190

FIGURE 52. Tensile Adhesion at -30°C, Sample 3



Adhesion Test Using T&T System - TAC Proposal for C&J Test

Test Type:	Tensile	Instron Corporation
Sample Identification:		Series IX Automated Materials Testing System 1.02C
Interface Type:	4200 Series	Test Date: 03 Dec 1995
Machine Parameters of Test:		Sample Type: ASTM
Sample rate (pts/sec):	5.00	Humidity (%): 50
Crosshead Speed (mm/min):	10.000	Temperature (deg. C): -30

Sample Description: 4

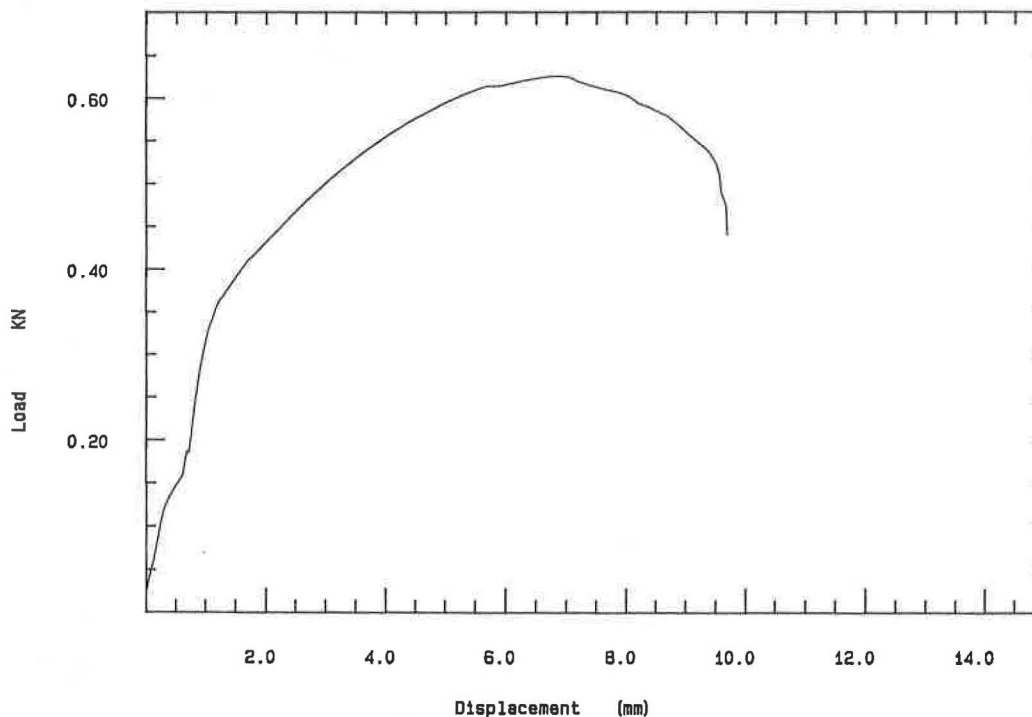
Dimensions:

	<u>Spec. 1</u>	<u>Spec. 2</u>	<u>Spec.3</u>	<u>Spec. 4</u>	<u>Spec. 5</u>
Diameter (mm)	25.400	25.400	25.400	25.400	25.400
Spec gauge len (mm)	25.416	25.478	28.058	28.399	26.228
Grip distance (mm)	125.000	125.000	125.000	125.000	125.000

Out of 5 specimens, 1 excluded.

Specimen No.	Toughness (J)	Peak Load (N)	Ratio of Toughness	Displacement at Max. Load (mm)	Maximum Displacement (mm)
Excluded	.1903	265.6	.0007	1.064	1.410
2	7.4120	692.3	.0107	9.812	13.180
3	4.7870	623.1	.0077	5.682	9.701
4	5.9540	630.6	.0094	9.191	12.060
Mean	6.7960	666.2	.0101	9.214	12.350
Std. Deviation	1.8370	46.8	.0021	2.682	2.021

Figure 53. Tensile Adhesion at -30°C, Sample 4



Adhesion Test Using T&T System - TAC Proposal for C&J Test

Test Type:	Tensile	Instron Corporation
Sample Identification:		Series IX Automated Materials Testing System 1.02C
Interface Type:	4200 Series	Test Date: 17 Dec 1995
Machine Parameters of Test:		Sample Type: ASTM
Sample rate (pts/sec):	5.00	Humidity (%): 50
Crosshead Speed (mm/min):	10.000	Temperature (deg. C): -30

Sample Description: 5

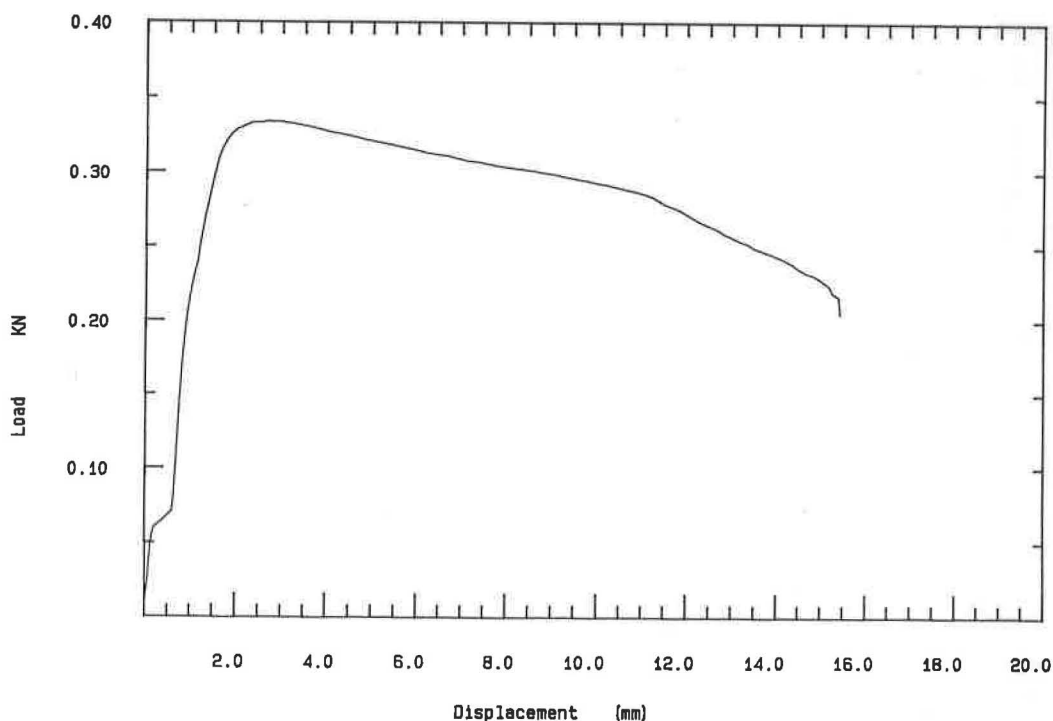
Dimensions:

	<u>Spec. 1</u>	<u>Spec. 2</u>	<u>Spec.3</u>	<u>Spec. 4</u>
Diameter (mm)	25.400	25.400	25.400	25.400
Spec gauge len (mm)	25.421	25.443	25.489	25.861
Grip distance (mm)	125.000	125.000	125.000	125.000

Out of 4 specimens, 0 excluded.

Specimen No.	Toughness (J)	Peak Load (N)	Ratio of Toughness	Displacement at Max. Load (mm)	Maximum Displacement (mm)
1	6.363	329.1	.0193	2.429	23.03
2	5.210	343.9	.0152	3.397	19.48
3	4.008	359.6	.0111	1.981	15.43
4	6.133	310.1	.0198	2.669	22.54
Mean	5.429	335.7	.0164	2.619	20.12
Std. Deviation	1.070	21.1	.0040	.592	3.50

FIGURE 54. Tensile Adhesion at -30°C, Sample 5



Adhesion Test Using T&T System - TAC Proposal for C&J Test

Test Type:	Tensile	Instron Corporation
Sample Identification:		Series IX Automated Materials Testing System 1.02C
Interface Type:	4200 Series	Test Date: 03 Dec 1995
Machine Parameters of Test:		Sample Type: ASTM
Sample rate (pts/sec):	5.00	Humidity (%): 50
Crosshead Speed (mm/min):	10.000	Temperature (deg. C): -30

Sample Description: 6

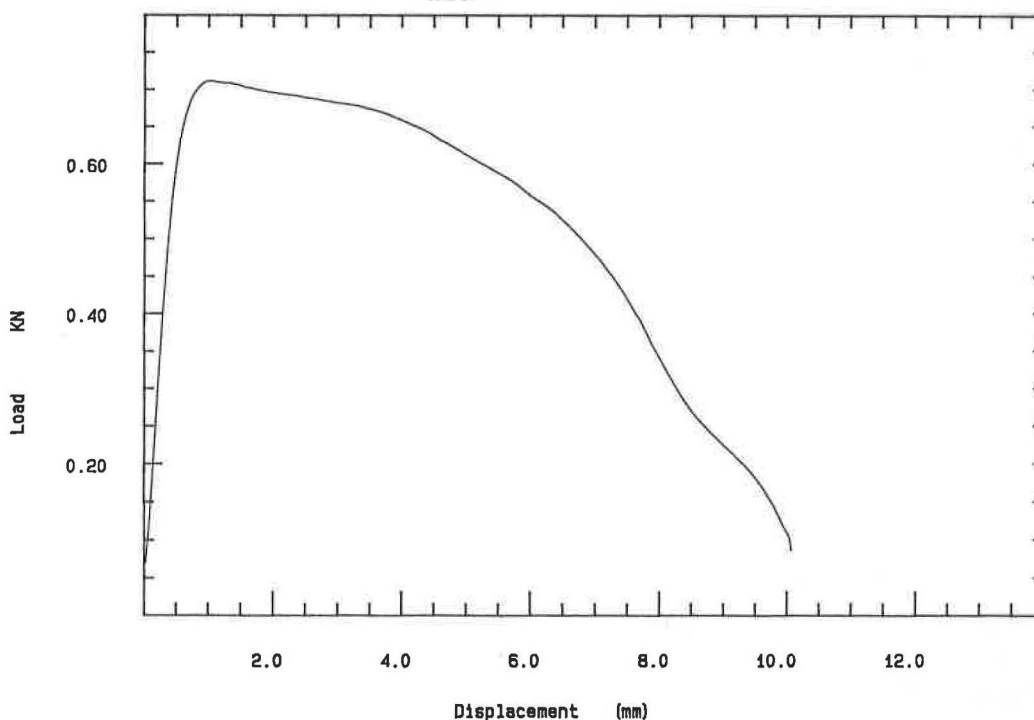
Dimensions:

	<u>Spec. 1</u>	<u>Spec. 2</u>	<u>Spec.3</u>	<u>Spec. 4</u>
Diameter (mm)	25.400	25.400	25.400	25.400
Spec gauge len (mm)	25.887	25.896	27.661	25.958
Grip distance (mm)	125.000	125.000	125.000	125.000

Out of 4 specimens, 0 excluded.

Specimen No.	Toughness (J)	Peak Load (N)	Ratio of Toughness	Displacement at Max. Load (mm)	Maximum Displacement (mm)
1	6.322	705.8	.0090	1.033	11.40
2	4.559	680.8	.0067	1.004	10.30
3	4.955	789.0	.0063	1.089	12.10
4	5.262	702.3	.0075	2.682	10.07
Mean	5.275	719.5	.0074	1.452	10.97
Std. Deviation	.755	47.6	.0012	.820	.95

FIGURE 55. Tensile Adhesion at -30°C, Sample 6



Adhesion Test Using T&T System - TAC Proposal for C&J Test

Test Type:	Tensile	Instron Corporation
Sample Identification:		Series IX Automated Materials Testing System 1.02C
Interface Type:	4200 Series	Test Date: 17 Dec 1995
Machine Parameters of Test:		Sample Type: ASTM
Sample rate (pts/sec):	5.00	Humidity (%): 50
Crosshead Speed (mm/min):	10.000	Temperature (deg. C): -30

Sample Description: 7

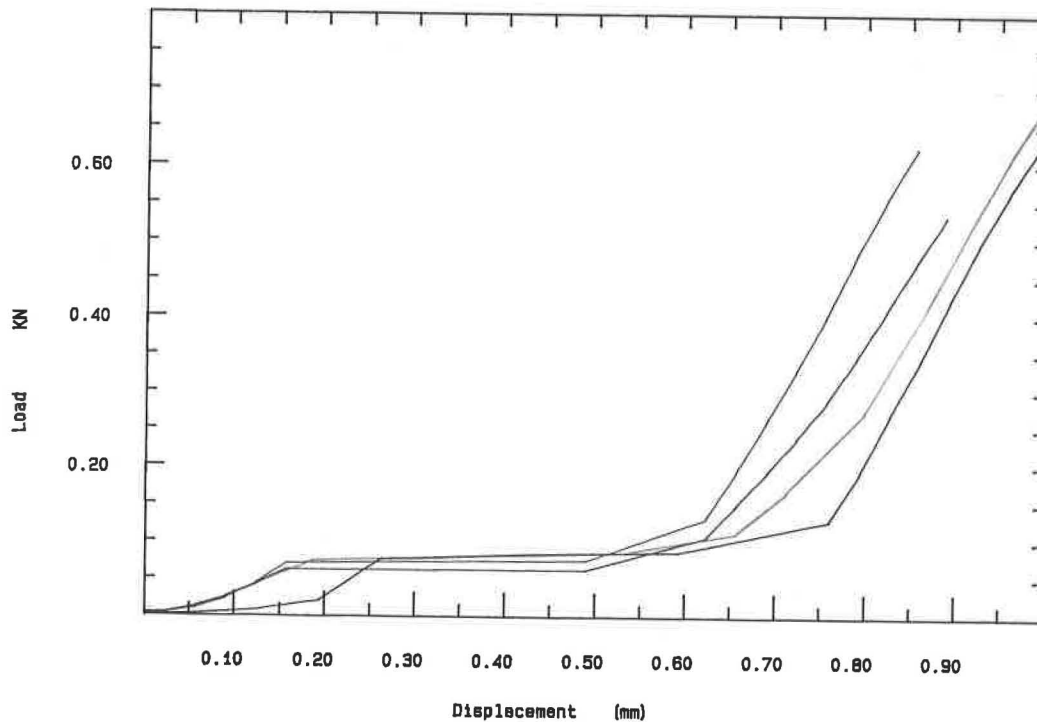
Dimensions:

	<u>Spec. 1</u>	<u>Spec. 2</u>	<u>Spec.3</u>	<u>Spec. 4</u>
Diameter (mm)	25.400	25.400	25.400	25.400
Spec gauge len (mm)	26.113	25.981	25.425	26.006
Grip distance (mm)	125.000	125.000	125.000	125.000

Out of 4 specimens, 0 excluded.

Specimen No.	Toughness (J)	Peak Load (N)	Ratio of Toughness	Displacement at Max. Load (mm)	Maximum Displacement (mm)
1	.1620	692.1	.0002	.3269	.3473
2	.1296	651.5	.0002	.2885	.2748
3	.2138	798.9	.0003	1.0650	1.0690
4	.1176	550.1	.0002	.3043	.2842
Mean	.1557	673.2	.0002	.4961	.4939
Std. Deviation	.0430	102.9	.0000	.3795	.3849

FIGURE 56. Tensile Adhesion at -30°C, Sample 7



Adhesion Test Using T&T System - TAC Proposal for C&J Test

Test Type:	Tensile	Instron Corporation
Sample Identification:		Series IX Automated Materials Testing System 1.02C
Interface Type:	4200 Series	Test Date: 14 JAN 1996
Machine Parameters of Test:		Sample Type: ASTM
Sample rate (pts/sec):	5.00	Humidity (%): 50
Crosshead Speed (mm/min):	10.000	Temperature (deg. C): -30

Sample Description: 8

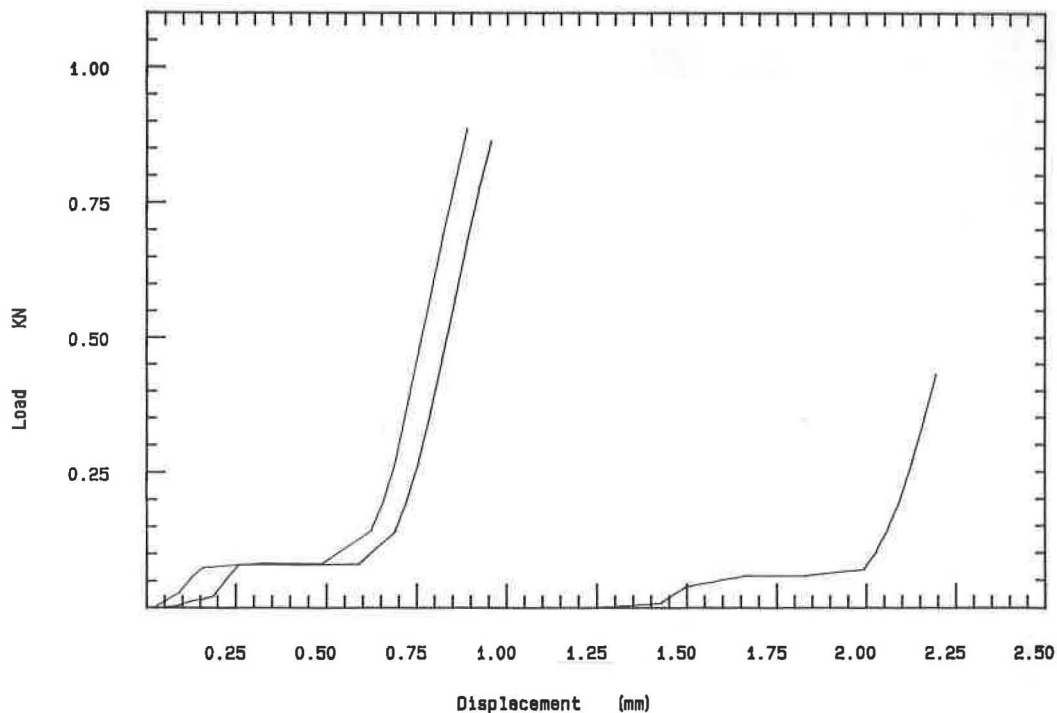
Dimensions:

	<u>Spec. 1</u>	<u>Spec. 2</u>	<u>Spec.3</u>	<u>Spec. 4</u>
Diameter (mm)	25.400	25.400	25.400	25.400
Spec gauge len (mm)	26.078	26.003	25.387	25.400
Grip distance (mm)	125.000	125.000	125.000	125.000

Out of 4 specimens, 1 excluded.

Specimen No.	Toughness (J)	Peak Load (N)	Ratio of Toughness	Displacement at Max. Load (mm)	Maximum Displacement (mm)
1	.1710	863.1	.0002	.2822	.2775
2	.1762	885.9	.0002	.2865	.2855
Excluded	.0698	228.5	.0003	.7432	.7404
4	.0771	477.9	.0002	2.2100	.7600
Mean	.1414	742.3	.0002	.9262	.4410
Std. Deviation	.0558	229.3	.0000	1.1120	1.1040

FIGURE 57. Tensile Adhesion at -30°C, Sample 8



Adhesion Test Using T&T System - TAC Proposal for C&J Test

Test Type:	Tensile	Instron Corporation
Sample Identification:		Series IX Automated Materials Testing System 1.02C
Interface Type:	4200 Series	Test Date: 17 DEC 1995
Machine Parameters of Test:		Sample Type: ASTM
Sample rate (pts/sec):	5.00	Humidity (%): 50
Crosshead Speed (mm/min):	10.000	Temperature (deg. C): -30

Sample Description: 9

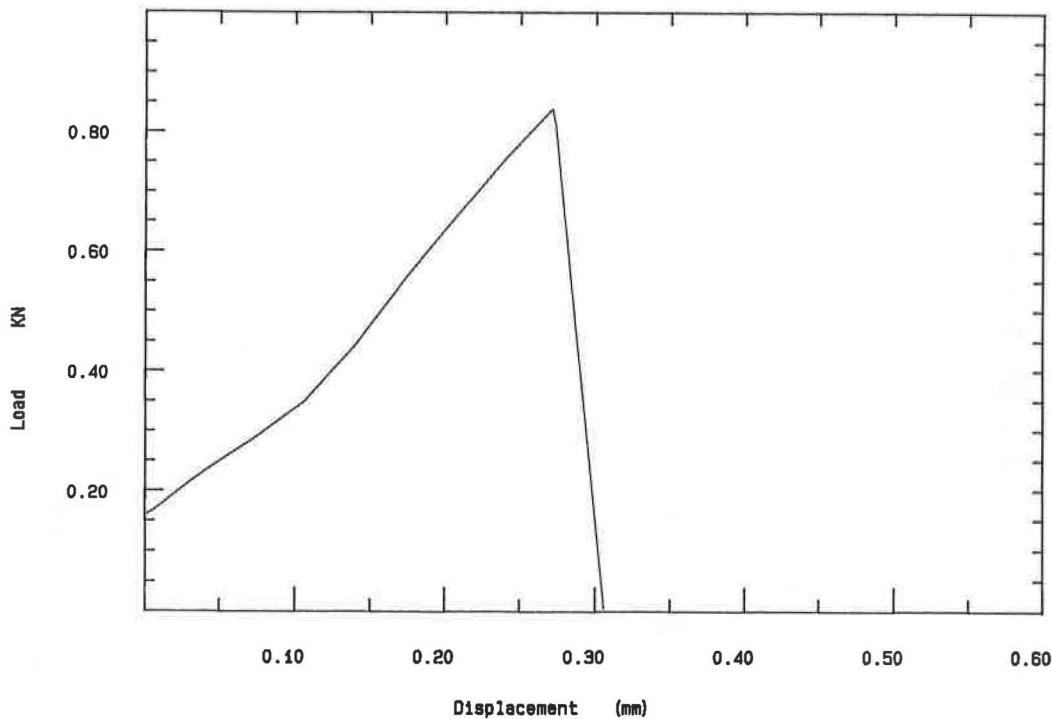
Dimensions:

	<u>Spec. 1</u>	<u>Spec. 2</u>	<u>Spec. 3</u>
Diameter (mm)	25.400	25.400	25.400
Spec gauge len (mm)	26.320	27.075	26.104
Grip distance (mm)	125.000	125.000	125.000

Out of 1 specimens, 0 excluded.

Specimen No.	Toughness (J)	Peak Load (N)	Ratio of Toughness	Displacement at Max. Load (mm)	Maximum Displacement (mm)
1	.1729	841.9	.0002	.2705	.3058
2	.1160	750.9	.0002	.2350	.2477
3	.1245	725.4	.0002	.2262	.2244
Mean	.1378	772.7	.0002	.4772	.3315
Std. Deviation	.0307	61.2	.0000	.3892	.0992

FIGURE 58. Tensile Adhesion at -30°C, Sample 9



Adhesion Test Using T&T System - TAC Proposal for C&J Test

Test Type:	Tensile	Instron Corporation
Sample Identification:		Series IX Automated Materials Testing System 1.02C
Interface Type:	4200 Series	Test Date: 16 DEC 1995
Machine Parameters of Test:		Sample Type: ASTM
Sample rate (pts/sec):	5.00	Humidity (%): 50
Crosshead Speed (mm/min):	10.000	Temperature (deg. C): -30

Sample Description: 10

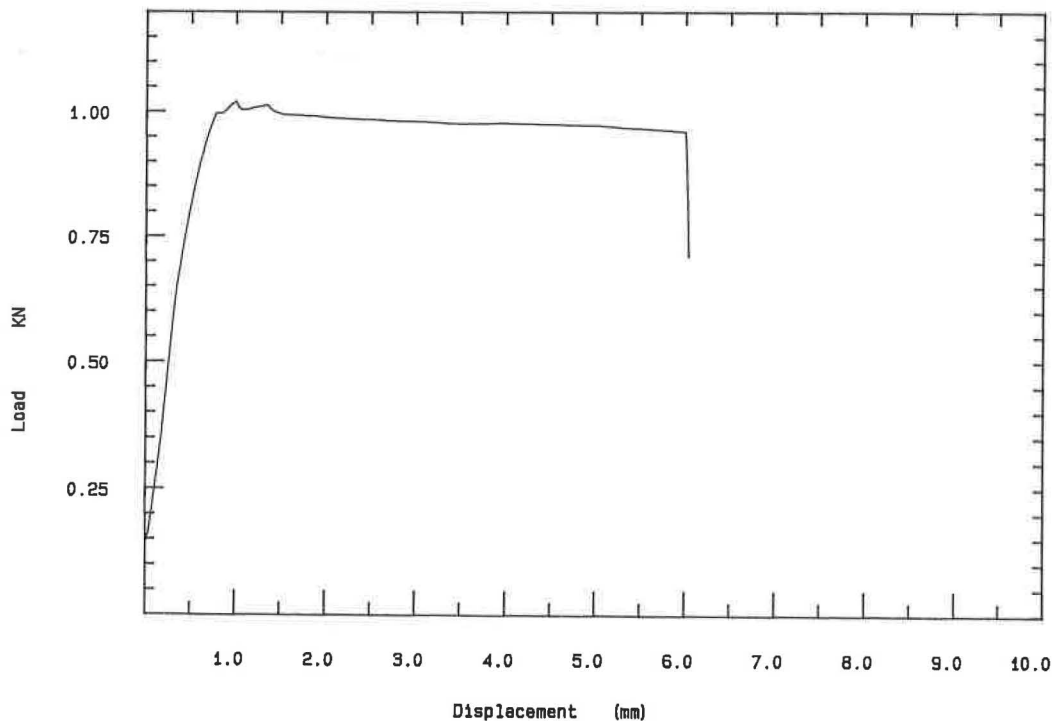
Dimensions:

	<u>Spec. 1</u>	<u>Spec. 2</u>	<u>Spec.3</u>	<u>Spec. 4</u>	<u>Spec. 5</u>
Diameter (mm)	25.400	25.400	25.400	25.400	25.400
Spec gauge len (mm)	27.645	29.943	26.045	26.134	25.993
Grip distance (mm)	125.000	125.000	125.000	125.000	125.000

Out of 5 specimens, 1 excluded.

Specimen No.	Toughness (J)	Peak Load (N)	Ratio of Toughness	Displacement at Max. Load (mm)	Maximum Displacement (mm)
1	6.370	1156.0	.0055	1.4250	6.045
2	21.690	1089.0	.0199	.8065	21.220
Excluded	1.158	1022.0	.0011	.9551	2.947
4	19.220	1031.0	.0186	18.1700	19.990
5	20.990	1050.0	.0200	21.8800	22.340
Mean	17.070	1082.0	.0160	10.5700	17.400
Std. Deviation	7.207	55.0	.0070	11.0200	7.628

FIGURE 59. Tensile Adhesion at -30°C, Sample 10



Adhesion Test Using T&T System - TAC Proposal for C&J Test

Test Type:	Tensile	Instron Corporation
Sample Identification:		Series IX Automated Materials Testing System 1.02C
Interface Type:	4200 Series	Test Date: 16 DEC 1995
Machine Parameters of Test:		Sample Type: ASTM
Sample rate (pts/sec):	5.00	Humidity (%): 50
Crosshead Speed (mm/min):	10.000	Temperature (deg. C): -30

Sample Description: 11

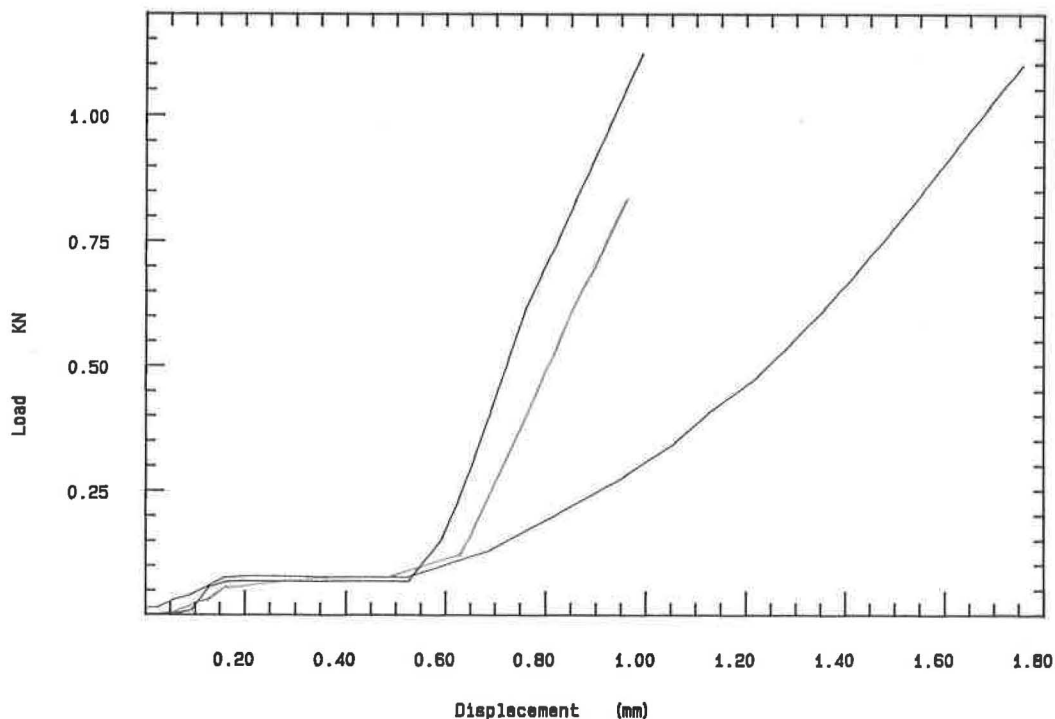
Dimensions:

	<u>Spec. 1</u>	<u>Spec. 2</u>	<u>Spec.3</u>
Diameter (mm)	25.400	25.400	25.400
Spec gauge len (mm)	25.949	26.281	25.992
Grip distance (mm)	125.000	125.000	125.000

Out of 3 specimens, 1 excluded.

Specimen No.	Toughness (J)	Peak Load (N)	Ratio of Toughness	Displacement at Max. Load (mm)	Maximum Displacement (mm)
1	.3017	1121.0	.0003	.4413	.4435
2	.7019	1186.0	.0006	.9493	.9423
3	.2009	850.5	.0002	.3784	.3687
Mean	.4015	1053.0	.0004	.5896	.5848
Std. Deviation	.2650	177.9	.0002	.3130	.3118

FIGURE 60. Tensile Adhesion at -30°C, Sample 11



Adhesion Test Using T&T System - TAC Proposal for C&J Test

Test Type:	Tensile	Instron Corporation
Sample Identification:		Series IX Automated Materials Testing System 1.02C
Interface Type:	4200 Series	Test Date: 14 JAN 1996
Machine Parameters of Test:		Sample Type: ASTM
Sample rate (pts/sec):	5.00	Humidity (%): 50
Crosshead Speed (mm/min):	10.000	Temperature (deg. C): -30

Sample Description: 12

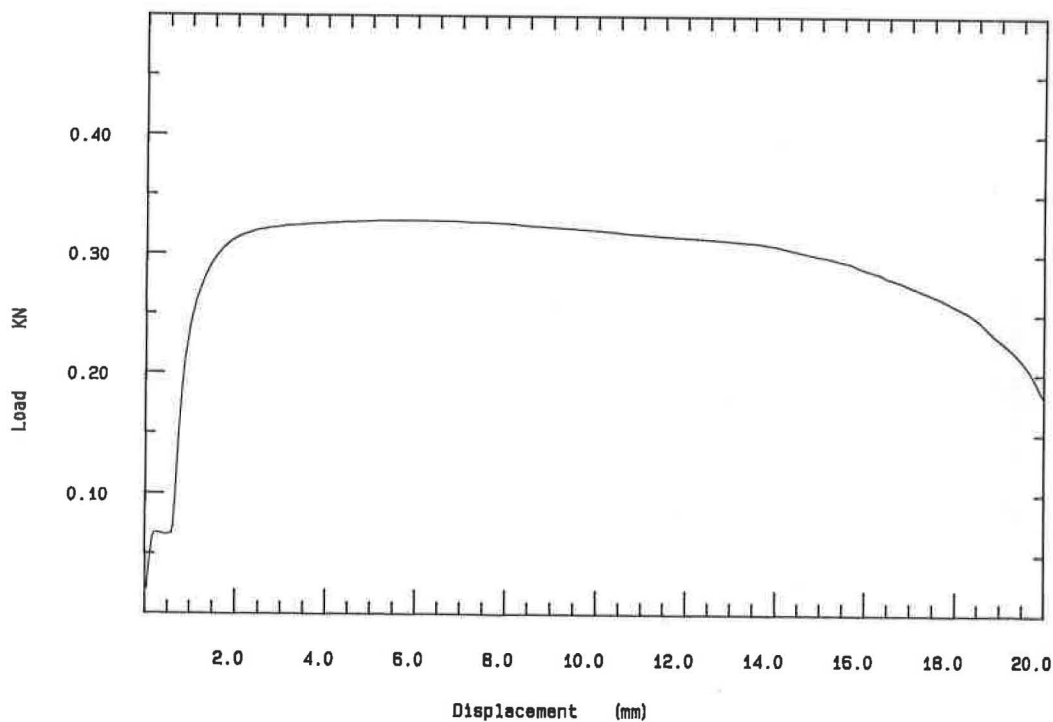
Dimensions:

	<u>Spec. 1</u>	<u>Spec. 2</u>	<u>Spec.3</u>	<u>Spec. 4</u>
Diameter (mm)	25.400	25.400	25.400	25.400
Spec gauge len (mm)	25.404	25.500	25.466	25.487
Grip distance (mm)	125.000	125.000	125.000	125.000

Out of 4 specimens, 0 excluded.

Specimen No.	Toughness (J)	Peak Load (N)	Ratio of Toughness	Displacement at Max. Load (mm)	Maximum Displacement (mm)
1	6.309	343.4	.0184	3.496	21.69
2	7.198	324.6	.0222	2.000	25.12
3	5.542	327.7	.0169	5.164	21.16
4	5.838	330.5	.0177	5.783	20.57
Mean	6.222	331.5	.0188	4.111	22.14
Std. Deviation	.723	8.3	.0023	1.707	2.04

FIGURE 61. Tensile Adhesion at -30°C, Sample 12



Adhesion Test Using T&T System - TAC Proposal for C&J Test

Test Type:	Tensile	Instron Corporation
Sample Identification:		Series IX Automated Materials Testing System 1.02C
Interface Type:	4200 Series	Test Date: 25 JAN 1996
Machine Parameters of Test:		Sample Type: ASTM
Sample rate (pts/sec):	5.00	Humidity (%): 50
Crosshead Speed (mm/min):	10.000	Temperature (deg. C): -30

Sample Description: 13

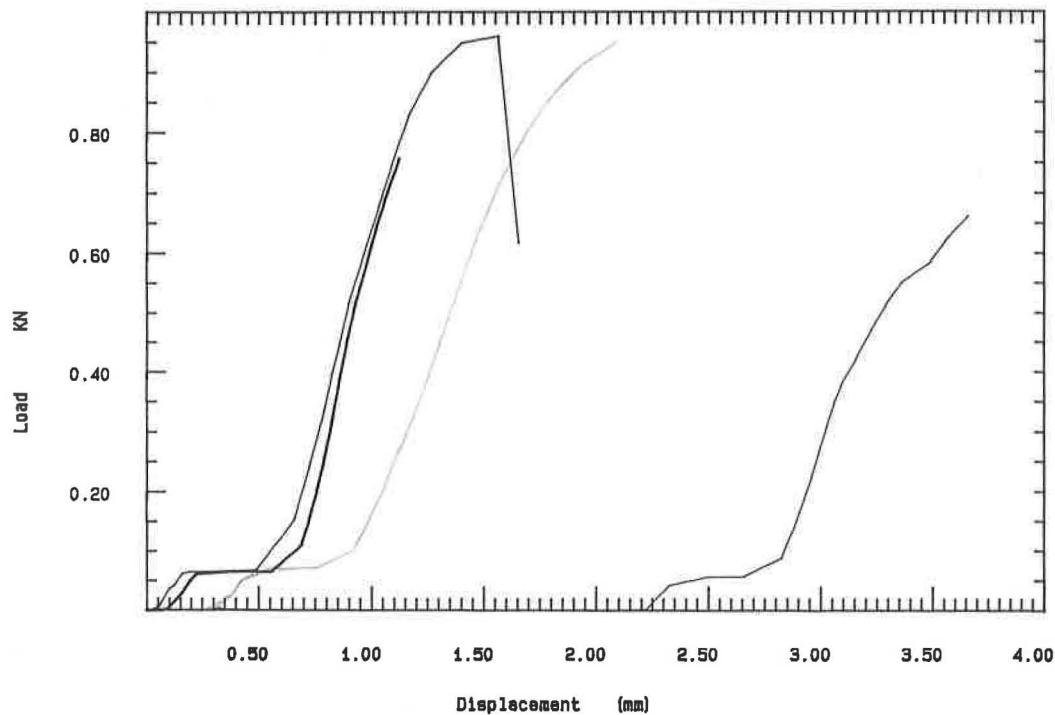
Dimensions:

	<u>Spec. 1</u>	<u>Spec. 2</u>	<u>Spec.3</u>	<u>Spec. 4</u>	<u>Spec. 5</u>
Diameter (mm)	25.400	25.400	25.400	25.400	25.400
Spec gauge len (mm)	25.985	26.059	26.289	28.179	26.062
Grip distance (mm)	125.000	125.000	125.000	125.000	125.000

Out of 5 specimens, 1 excluded.

Specimen No.	Toughness (J)	Peak Load (N)	Ratio of Toughness	Displacement at Max. Load (mm)	Maximum Displacement (mm)
1	.6731	961.7	.0007	.9753	1.0710
Excluded	.2129	480.0	.0004	.6113	.5980
3	.7420	971.1	.0008	1.3310	1.2010
4	.4008	665.8	.0006	.8813	.8777
5	.2367	759.2	.0003	.4578	.4622
Mean	.5132	839.4	.0006	.9112	.9029
Std. Deviation	.2359	151.5	.0002	.3589	.3224

FIGURE 62. Tensile Adhesion at -30°C, Sample 13



Adhesion Test Using T&T System - TAC Proposal for C&J Test

Test Type:	Tensile	Instron Corporation
Sample Identification:		Series IX Automated Materials Testing System 1.02C
Interface Type:	4200 Series	Test Date: 03 DEC 1995
Machine Parameters of Test:		Sample Type: ASTM
Sample rate (pts/sec):	5.00	Humidity (%): 50
Crosshead Speed (mm/min):	10.000	Temperature (deg. C): -30

Sample Description: 14

Dimensions:

	<u>Spec. 1</u>	<u>Spec. 2</u>	<u>Spec.3</u>	<u>Spec. 4</u>
Diameter (mm)	25.400	25.400	25.400	25.400
Spec gauge len (mm)	25.405	29.438	26.086	27.748
Grip distance (mm)	125.000	125.000	125.000	125.000

Out of 4 specimens, 0 excluded.

Specimen No.	Toughness (J)	Peak Load (N)	Ratio of Toughness	Displacement at Max. Load (mm)	Maximum Displacement (mm)
1	11.16	338.5	.0330	3.875	49.99
2	13.09	341.6	.0383	5.352	56.89
3	16.77	463.4	.0362	2.884	73.07
4	16.40	450.2	.0364	2.962	96.01
Mean	14.35	398.4	.0360	3.768	68.99
Std. Deviation	2.70	67.6	.0022	1.148	20.45

FIGURE 63. Tensile Adhesion at -30°C, Sample 14

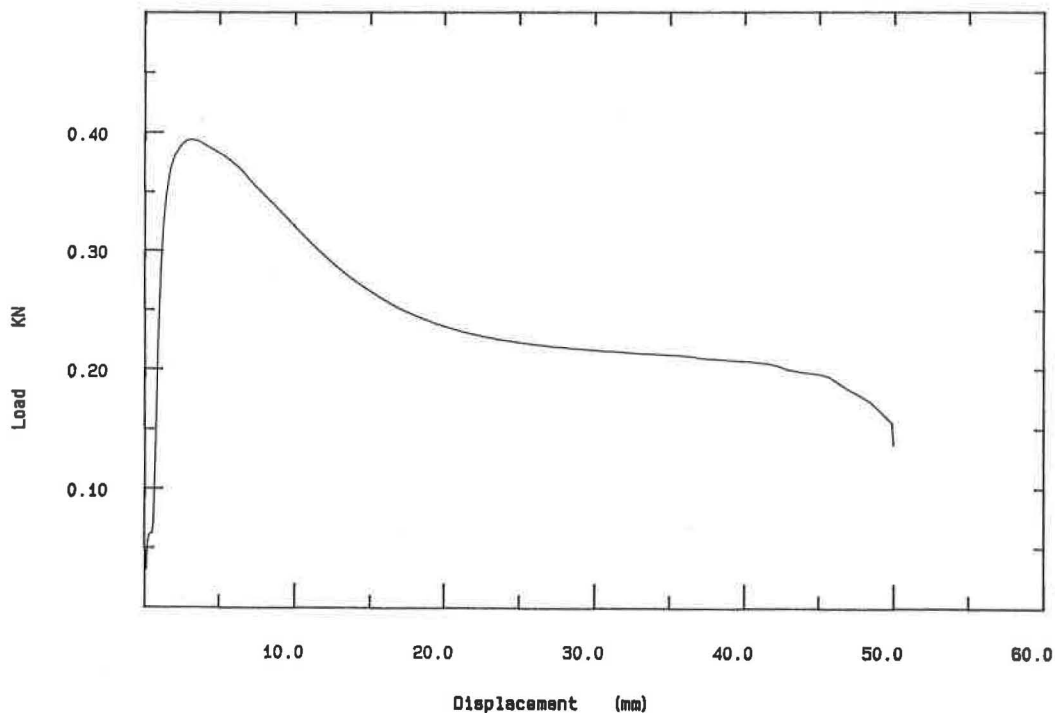


FIGURE 64. Peak Load of Crack Sealants in Stress Relaxation Test

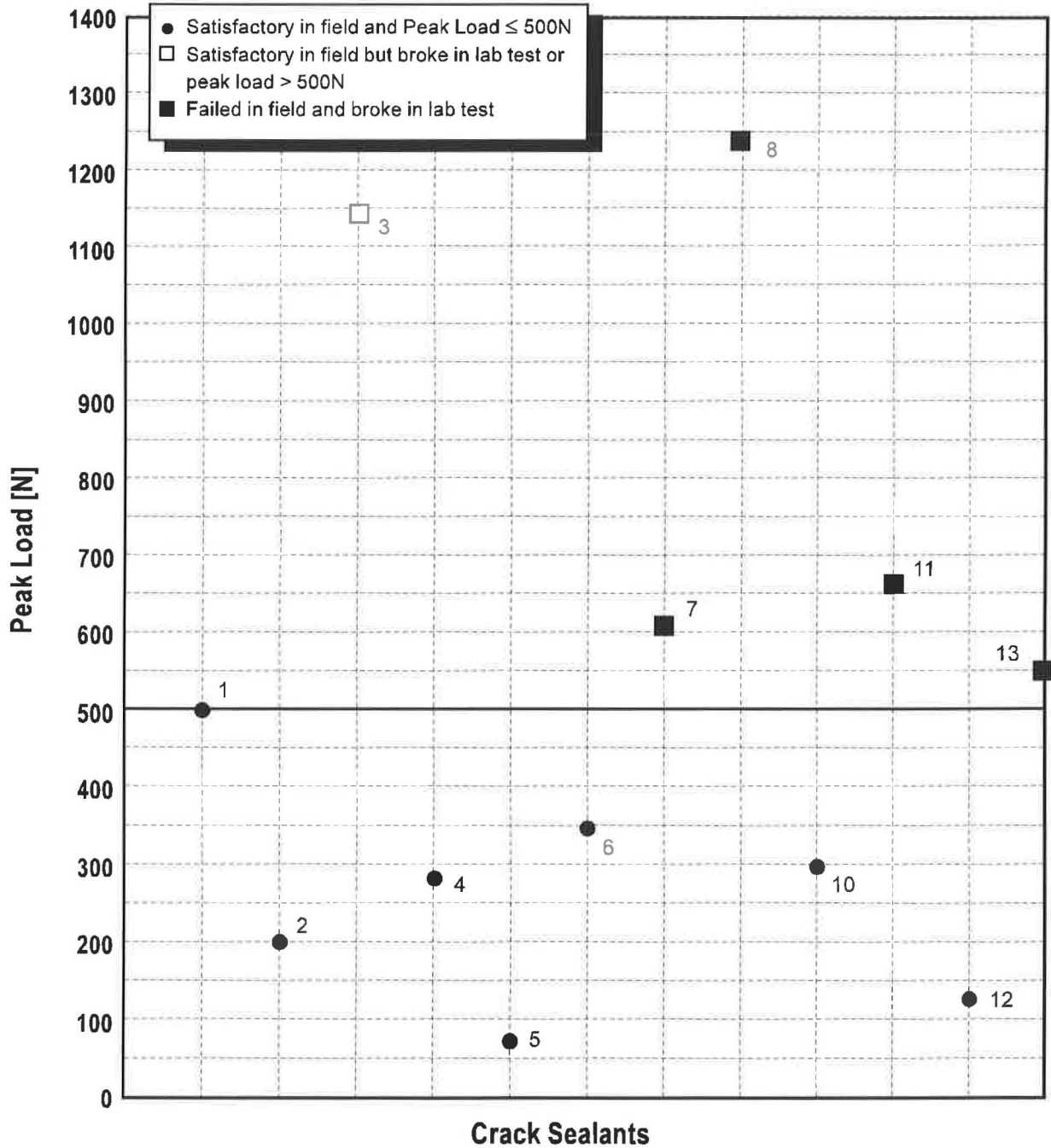


FIGURE 65. Maximum Extension at Crack Sealant Debonding in Tensile Adhesion Test

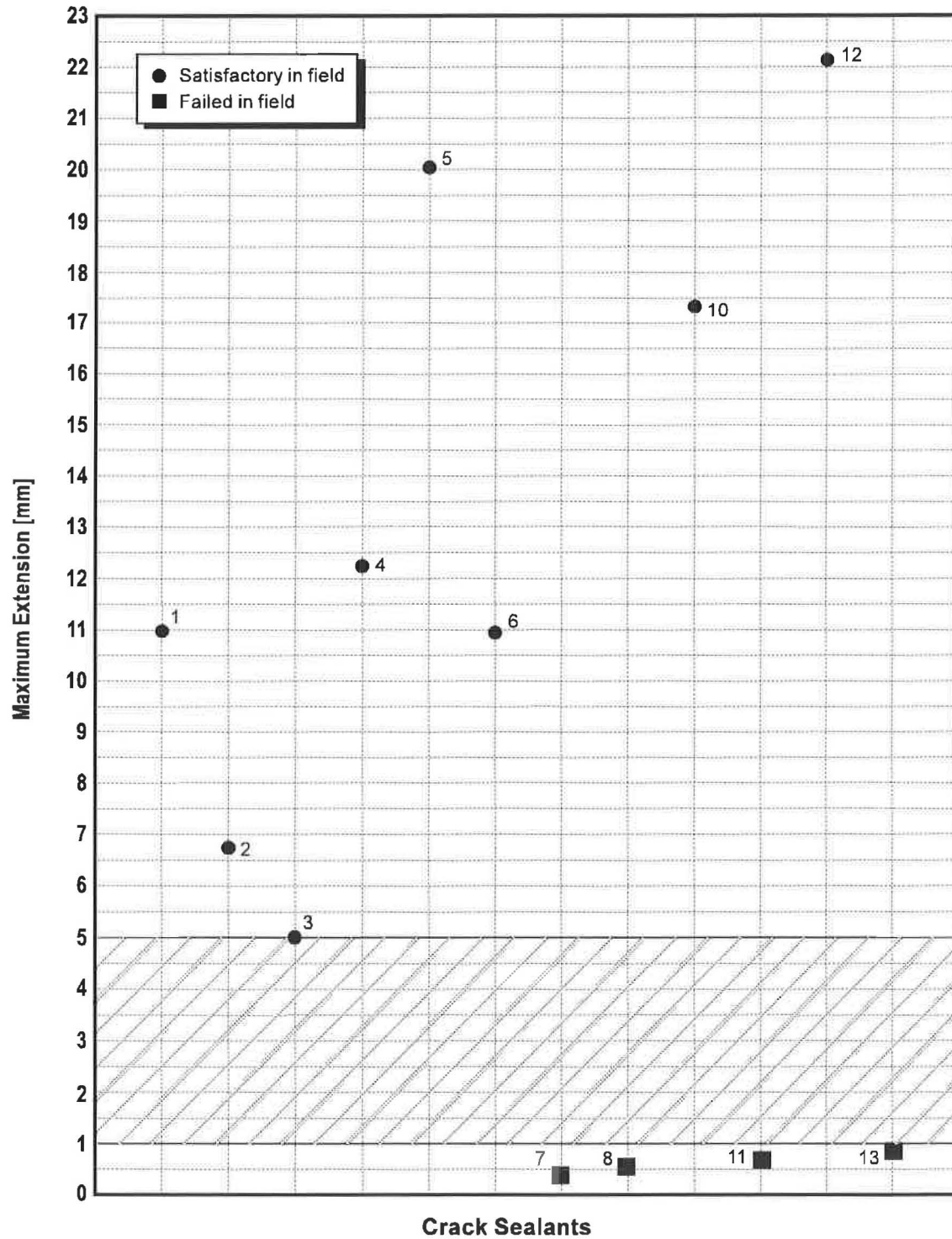
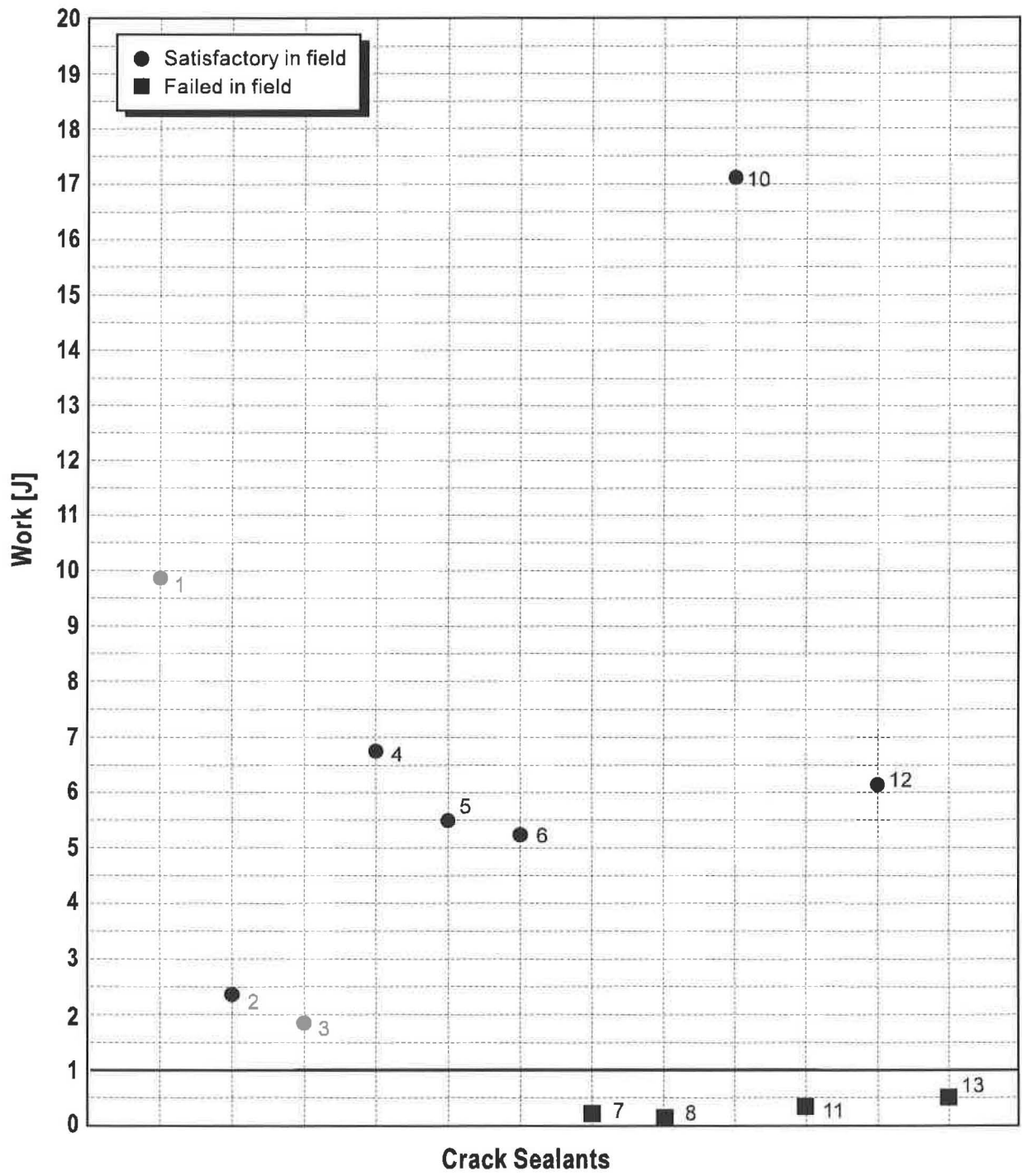


FIGURE 66. Total Work Necessary for Crack Sealant Debonding



Appendix:
**Rheological Analysis of the
Stress Relaxation Test and
Behaviour of Crack Sealants**

RHEOLOGICAL ANALYSIS OF THE STRESS RELAXATION TEST FOR CRACK SEALING MATERIALS

INTRODUCTION

Conjecture that faster stress relaxation in sealants leads to a higher bonding integrity to the crack wall has been proposed. A description of the experimental setup for the stress relaxation test is given in Testing Protocol. Theoretical analysis of the relaxation experiment described in Testing Protocol is given here.

EXTENSIONAL FLOW

The stress relaxation test described in [1] is an elongational flow for which the velocity field v_i , in Cartesian coordinates (x_i), has the following form:

$$v_1 = \dot{\epsilon} x_1, \quad v_2 = -\frac{1}{2} \dot{\epsilon} x_2, \quad v_3 = -\frac{1}{2} \dot{\epsilon} x_3 \quad (1)$$

where $\dot{\epsilon}(t)$ is the elongational rate, generally depending on time t , and v_i are Cartesian components of the velocity field $\underline{v}(\underline{x})$.

Solving equation (1) one obtains the components of the position vector \underline{x} :

$$\begin{aligned} x_1 &= x_1^0 \exp \left(\int_0^t \dot{\epsilon}(t') dt' \right) \\ x_2 &= x_2^0 \exp \left(-\frac{1}{2} \int_0^t \dot{\epsilon}(t') dt' \right) \\ x_3 &= x_3^0 \exp \left(-\frac{1}{2} \int_0^t \dot{\epsilon}(t') dt' \right) \end{aligned} \quad (2)$$

The form of the stress tensor in elongation flows is

$$\underline{T} = -p\underline{I} + \underline{\tau}, \quad T_{ij} = \begin{pmatrix} -p + \tau_{11} & 0 & 0 \\ 0 & -p + \tau_{22} & 0 \\ 0 & 0 & -p + \tau_{33} \end{pmatrix} \quad (3)$$

where \underline{T} is the stress tensor, p is the pressure, and $\underline{\tau}$ represents the extra-stress tensor.

In incompressible materials there are only two independent normal stress differences, $\tau_{11} - \tau_{22}$, and $\tau_{22} - \tau_{33}$.

In the studied elongational flow the directions 2 and 3 are indistinguishable, i.e. $\tau_{22} = \tau_{33}$. Thus only one normal stress difference ($\tau_{11} - \tau_{22}$) can be determined.

Define the pressure p as,

$$p = -\frac{1}{3}(T_{11} + T_{22} + T_{33}) \quad (4)$$

and assume (as is the case of the studied experiment) that there are no lateral forces, i.e.,

$$T_{22} = 0, T_{33} = 0 \quad (5)$$

Then,

$$p = -\frac{1}{3}T_{11} = -\frac{1}{3}(-p + \tau_{11})$$

and

$$p = -\frac{\tau_{11}}{2} \quad (6)$$

Using (6), and the fact that $T_{22} = 0$, i.e. $0 = -p + \tau_{22}$, one also has

$$T_{11} = -p + \tau_{11} = \frac{3}{2}\tau_{11} \quad (7)$$

and

$$\tau_{11} - \tau_{22} = \tau_{11} - p = \tau_{11} + \frac{\tau_{11}}{2} = \frac{3}{2}\tau_{11} \quad (8)$$

The tested sample had the cylindrical shape with the approximate diameter 25×10^{-3} m, and height also approximately 25×10^{-3} m. Thus it is natural to use the cylindrical coordinates $\{r, \phi, z\}$. Because in this coordinate system $v_\phi = 0$, then the continuity equation has the form

$$\frac{\partial v_z}{\partial t} + \frac{1}{r} \frac{\partial}{\partial r}(rv_r) = 0 \quad (9)$$

From the last equation it follows that

$$\frac{\partial v_z}{\partial t} = \text{const.} = -\frac{1}{r} \frac{\partial}{\partial r}(rv_r) \quad (10)$$

The appropriate boundary conditions are

$$v_z|_{z=0} = 0, v_z|_{z=L(t)} = U, v_r|_{r=0} = 0 \quad (11)$$

Here, L is the length of the tested sample, and U is the speed of the horizontal; plane $z = L(t)$.

The solution of the boundary value problem (10) and (11) is

$$v_z = \frac{U}{L}z, \quad v_r = -\frac{Ur}{2L} \quad (12)$$

The elongational deformation of the tested cylindrical sample does not depend on angle y , thus one can return to Cartesian coordinates, i.e.,

$$\begin{aligned} x_1 &= z, \quad x_2 = r, \quad x_3 = r \\ v_1 &= v_z, \quad v_2 = v_r, \quad v_3 = v_r \end{aligned} \quad (13)$$

It follows from (12) that

$$v_1 = \frac{U}{L}x_1, \quad v_2 = -\frac{U}{2L}x_2, \quad v_3 = -\frac{U}{2L}x_3 \quad (14)$$

which is consistent with equation (1).

If the initial length of the sample (direction $x_1 = z$) is L_0 then the length at time t is given as

$$L(t) = L_0 + \int_0^t U(t') dt' \quad (15)$$

where $v_z|_{z=L} = U$

Assuming that the volume of the sample is conserved thus the cross section $A(t)$ of the sample satisfies the following equation:

$$A(t)L(t) = A_0L_0 \quad (16)$$

where A_0 and L_0 are the original cross-section and length, respectively. The elongation of the sample can be described via the following function: $\alpha(t)$

$$\alpha(t) = L(t)/L_0 = A_0/A(t) \quad (17)$$

The elongational rate $\dot{\epsilon}$ can be calculated as follows (see (1)):

$$\dot{\epsilon} = \frac{\partial v_1}{\partial x_1} = \frac{U}{L} = \frac{1}{L} \left(\frac{\partial L}{\partial t} \right) = \frac{1}{\alpha} \left(\frac{d\alpha}{dt} \right) \quad (18)$$

It is clear from (8) that the elongational deformation with constant speed, $U = \text{const.}$, is not the flow with constant rate of elongation. Thus the tests performed and discussed in (1) are nonstandard and one is not

able to define and investigate the known material functions, (2). All of these functions are defined only for $\epsilon = \text{const}$. Notwithstanding these difficulties we proceed with the analysis of the stress relaxation tests. In this test the constant speed U was used, i.e.

$$U = U_0 \quad (19)$$

and

$$\dot{\epsilon}(t) = \frac{U_0}{L(t)} = \frac{U_0}{L_0} \frac{1}{\alpha(t)} \quad (20)$$

The following values of the relaxation test parameters have been used:

$$U_0 = \frac{1}{6} 10^{-4} \text{ s}^{-1}$$

$$L_0 = 25 \times 10^{-3} \text{ m} \quad (21)$$

$$A_0 = 490.874 \times 10^{-6} \text{ m}^2$$

The total time, t_e , during which the elongational flow was held was

$$t_e = 750 \text{ s} \quad (22)$$

Thus,

$$\frac{U_0}{L_0} = \frac{1}{1500} \text{ s}^{-1} \quad (23)$$

and

$$\alpha(t) = \frac{L_0 + U_0 t}{L_0} = 1 + \frac{t}{1500} \quad (24)$$

Also

$$\dot{\epsilon}(t) = \frac{U_0}{L_0} \frac{1}{\alpha(t)} = \frac{1}{1500 + t}, \quad t \in (0, t_e) \quad (25)$$

It follows from (21), that

$$\dot{\epsilon}(t) = \frac{d}{dt} \ln(1500 + t) = \frac{d}{dt} \ln \alpha(t) \quad (26)$$

Because $\alpha(t)$ is increasing on the interval $(0, 750)$, the elongational rate is decreasing there. Thus the studied experiment is not an experiment with constant elongational rate.

The total elongation, up to time t_e , is

$$\epsilon(O, t_e) = \int_0^{t_e} \dot{\epsilon}(t) dt = \ln\left(1 + \frac{1}{2}\right) - \ln(1) = \ln \frac{3}{2} \quad (27)$$

Now it is possible to write Eq. (2) in terms of $\alpha(t)$. Since $\dot{\epsilon}(t) = d \ln \alpha(t)/dt$ and $\alpha(0) = 1$, the Eq. (2) have the form

$$\begin{aligned} x_1 &= x_1^0 e^{\ln \alpha(t)} = x_1^0 \alpha(t) \\ x_2 &= x_2^0 e^{-\frac{1}{2} \ln \alpha(t)} = x_2^0 \alpha^{-\frac{1}{2}}(t) \\ x_3 &= x_3^0 e^{-\frac{1}{2} \ln \alpha(t)} = x_3^0 \alpha^{-\frac{1}{2}}(t) \end{aligned} \quad (28)$$

Defining the displacement function

$$\xi = \underline{x} - \underline{x}^0, \quad \xi_i = x_i - x_i^0 \quad (29)$$

one can calculate the deformation tensor (infinitesimal)

$$E_{ik} = \frac{1}{2} \left[\frac{\partial \xi_i}{\partial x_k} + \frac{\partial \xi_k}{\partial x_i} \right] \quad (30)$$

i.e.,

$$E_{ik} = \begin{pmatrix} \alpha - 1 & 0 & 0 \\ 0 & \alpha^{\frac{1}{2}-1} & \\ 0 & 0 & \alpha^{\frac{1}{2}-1} \end{pmatrix} \quad (31)$$

The elementary work, dW , of deformation is given as

$$dW = T_{ik} dE_{ik} = \text{tr}(\underline{T} \cdot d\underline{E}) \quad (32)$$

For studied experiment there are no lateral forces, thus $T_{22} = T_{33} = 0$, and it follows from (32)

$$dW = T_{11}d \quad (33)$$

The total work, W , of the extensional deformation (during the first 750s of the experiment) is then,

$$W = \int_1^{1.5} T_{11}d \quad (34)$$

As is seen from the last equation, one can calculate the work of deformation which brings the cylindrical sample to its 3/2 length in 750 seconds.

The axial force, F , which is measured, is given as

$$F = T_{11}A$$

Since $A = A_0/\alpha$ we have

$$F(\alpha) = T_{11}(\alpha)A_0/\alpha \quad (35)$$

and the axial stress T_{11} is given as

$$T_{11}(\alpha) = \frac{\alpha F(\alpha)}{A_0} \quad (36)$$

Then the deformation work (density of the deformation energy) per unit volume of the 50% extension is represented as follows:

$$W_{50\%} = \int_1^{1.5} \frac{\alpha F(\alpha)}{A_0} dx \quad (37)$$

RELAXATION

The elongational flow, sustained during the first 750 s, ends with the following values of parameters:

$$t_e = 750 \text{ s}$$

$$A_{750} = A_0 | \alpha(t_e) = \frac{2}{3} A_0 \quad (38)$$

$$A_0 = 490.874 \times 10^{-6} \text{ m}^2$$

Using (7), the extra-stress component τ_{11} is given as

$$\tau_{11} = \frac{2}{3} T_{11} = \frac{2}{3} \frac{F}{A} \quad (39)$$

Then for $t \in (0, 750)$

$$\tau_{11} = \frac{2}{3} F(t) \left(1 + \frac{t}{1500} \right) \frac{10^6}{\pi (12.5)^2} \quad (40)$$

and for $t \geq 750$

$$\tau_{11} = \frac{2}{3} \frac{F(t)}{2/3 A_0} = \frac{F(t) 10^6}{\pi (12.5)^2} \quad (41)$$

In the studied experiment the elongational rate $\dot{\epsilon}(t)$ has the form (see (25))

$$\dot{\epsilon}(t) = \frac{1}{1500 + t} [H(t) - H(t - 750)] \quad (42)$$

where H represents the Heaviside function.

If the studied sealants can be described as general viscoelastic materials, the following constitutive equation would apply to them, (1):

$$\tau_{ij}(t) = \int_{-\infty}^t E(t-t^{-1}) D_{ij}(t') dt' \quad (43)$$

where E represents a relaxation function, and D_{ij} is the rate of deformation tensor.

In our case $D_{ij}(t)$ has the form

$$D_{ij}(t) = \dot{\epsilon}(t) \begin{pmatrix} 2 & 0 & 0 \\ 0 & -1 & 0 \\ 0 & 0 & -1 \end{pmatrix} = \begin{pmatrix} 2 & 0 & 0 \\ 0 & -1 & 0 \\ 0 & 0 & -1 \end{pmatrix} \frac{1}{1500 + t} [H(t) - H(t - 750)] \quad (44)$$

Substituting (44) into the constitutive equation (43) one obtains

$$\tau_{ij}(t) = \begin{pmatrix} 2 & 0 & 0 \\ 0 & -1 & 0 \\ 0 & 0 & -1 \end{pmatrix} \left\{ \int_0^t \frac{E(t-t')}{1500+t'} dt' - \int_{750}^t \frac{E(t-t')}{1500+t'} dt' \right\} \quad (45)$$

$$t \geq 0$$

Substitution

$$1500 + t = \tau \quad (46)$$

yields

$$\tau_{ij}(t) = \begin{pmatrix} 2 & 0 & 0 \\ 0 & -1 & 0 \\ 0 & 0 & -1 \end{pmatrix} \left\{ \int_{1500}^{1500+t} \frac{E(t-\tau+1500)}{\tau} d\tau - \int_{2250}^{t+1500} \frac{E(t-\tau+(500))}{\tau} d\tau \right\} \quad (47)$$

Denote

$$t + 1500 = \sigma \quad (48)$$

then (47) yields

$$\begin{aligned} \tau_{ij}(\sigma - 1500) &= \begin{pmatrix} 2 & 0 & 0 \\ 0 & -1 & 0 \\ 0 & 0 & -1 \end{pmatrix} \left\{ \int_{1500}^{\sigma} \frac{E(\sigma - \tau)}{\tau} d\tau - \int_{2250}^{\sigma} \frac{E(\sigma - \tau)}{\tau} d\tau \right\} \\ &= \begin{pmatrix} 2 & 0 & 0 \\ 0 & -1 & 0 \\ 0 & 0 & -1 \end{pmatrix} \int_{1500}^{2250} \frac{E(\sigma - \tau)}{\tau} d\tau \end{aligned} \quad (49)$$

Since

$$\frac{\partial E}{\partial \sigma} = - \frac{\partial E}{\partial \tau} \quad (50)$$

Eq. (49) yields

$$\frac{\partial \tau_{ij}(\sigma - 1500)}{\partial \tau} = - \begin{pmatrix} 2 & 0 & 0 \\ 0 & -1 & 0 \\ 0 & 0 & -1 \end{pmatrix} \int_{1500}^{2250} \frac{1}{\tau} \frac{\partial E(\sigma - \tau)}{\partial \tau} d\tau \quad (51)$$

Here again one can see the problem with the experiments with variable elongational rate. If the experiment is set up in such a way that

$$U = \beta L_0 e^{\beta t}, \quad \beta = \text{const.} \quad (52)$$

the elongational rate ($\dot{\epsilon} = U/L = \beta$) would be constant and one can easily solve (51).

Unfortunately, this is not the case and we decided that instead of looking for a constitutive equation of the tested materials, we will try to describe the component τ_{11} of the extra-stress tensor in a simple mathematical form.

Using the measured values of the axial force $F(t)$, (1), one can calculate $\tau_{11}(t)$ via (40) and (41). We have found that obtained values of $\tau_{11}(t)$ can be fitted, for most of the samples, to the following function:

$$\tau_{11}(t) = \begin{cases} a \exp((t/\lambda)^b), & t < 750 \\ c \exp(-(t - 750)/\lambda)^d, & t \geq 750 \end{cases} \quad (53)$$

where a , b , c , d and λ are constants.

The first part of the function (53) describes the evolution of the stress during the elongation. This process

is stopped after the time interval of 750s, and the relaxation process immediately follows. It is clear that during such an experiment the sample is not in equilibrium. The second part of (53) describes the stress relaxation process. The rate of relaxation is given by the derivative of (53)₂, i.e.

$$\frac{-cd}{\lambda} \left(\frac{t-750}{\lambda} \right)^{d-1} \cdot \exp \left(- \left(\frac{t-750}{\lambda} \right)^d \right) \quad (54)$$

Thus the rate of stress relaxation is determined by the parameters c , d and λ .

Dividing $\tau_{11}(t)$ by $\tau_{11\max}$ one can compare the reduced curves $\tau_{11}/\tau_{11\max}$ of the studied samples.

In total, 46 samples have been analyzed by fitting the stretched exponential function to the $\tau_{11}(t)/\tau_{11}(\max)$.

$$\frac{\tau_{11}(t)}{\tau_{11}(\max)} = \begin{cases} a \exp [(t/\lambda)^b], & t < 750 \\ c \exp [-(t-750/\lambda)^d], & t \geq 750 \end{cases}$$

The results are summarized in Tables 1 through 3 and Figures 1 through 43. Table 1 summarizes the data for crack sealants tested in Phase II of the project. Table 2 summarizes crack sealants tested in Phase III of the project. Table 3 ranks all crack sealants according to increasing area under the relaxation part of the stress relaxation curve. Figures 1 through 35 are also organized according to the same pattern. Figures 36 through 43 show stress relaxation curves with pattern suggesting that the crack sealants broke during the extension period. The rate of relaxation can be effectively estimated by the area (A_1) beneath the relaxation part of the curve, $\tau_{11}(t)/\tau_{11}(\max)$.

Similarly, the extension part of the experiment can be effectively quantified by the area (A_2) beneath the extensional part of the curve $\tau_{11}(t)/\tau_{11}(\max)$.

One can say that of the tested samples follow the stretched exponential relaxation, with the exception of two groups of materials. The first group contains the following crack sealants: nos 3, 7, 9, 13. The crack sealant nos. 3 and 9 were most probably broken during the extension, as seen from the plots of $\tau_{11}(t)$ vs. t . Crack sealants nos. 7 and 13 exhibit a typical overshoot of τ_{11} during the elongation, which is followed by a continuous decrease of τ_{11} . Such behaviour might be attributed to the flow of these specimens. The fit of the curves $\tau_{11}(t)$ has not been attempted because of the unknown nature of the mentioned effects.

The second group, where the stretched exponential function slightly underestimates the long time behaviour, is formed by crack sealants: nos. 1, 2, 4, 6, and 10 (both from first and second group of the tested crack sealants). From these sample 2382, which corresponds with sample 2, exhibits a possible second relaxation region starting at $t \sim 2500s$.

The relaxation part of the reduced extra stress τ_{11} , for the rest of the crack sealants, is very well described by the stretched exponential function. Crack sealants, sample 2383 and sample 2448, exhibit a different extensional behaviour. In sample 2383 the decrease $\tau_{11}/\tau_{11}(\max)$ has recovered a little until the elongation is stopped and relaxation begins. In sample 2448, the maximum of $\tau_{11}/\tau_{11}(\max)$ is attained much more slowly during the extension. However, no recovery is observed. The initial fast growth of $\tau_{11}/\tau_{11}(\max)$ followed by the slower growth is observed in the rest of the crack sealants.

CONCLUSION

The rheological analysis of the behavior of modified crack and joint sealants as manifested through the proposed test of stress relaxation was performed.

As the results confirm, the modified crack and joint sealants are rheologically very complex materials which may manifest very different rheological behavior while submitted to the same testing conditions. Majority of them in this test behave like linear viscoelastic materials. Others, however, at the same conditions behave as non-linear viscoelastic materials, which might have been in the report (1) described as "ductile" behavior. These differences are either due to a difference in internal structure of tested crack and joint sealants, or even the rearrangement of the structure under strain.

The idea behind the tests proposed in the progress reports on the project was that in the realm of lower service temperatures, the important behavior of crack and joint sealant is (1) the willingness to relax the stresses caused by the contraction of the crack and joint sealant and opening of the crack through flow and (2) the capability of the crack and joint sealant to adhere to the wall.

When the analysis of results was made for the progress reports, in the test for stress relaxation a consideration was made about the importance of the peak load after 12.5 min of the test, that means, when crack and joint sealant was elongated by 50% and the relaxation period began.

The rheological analysis of the tests indicates, that since during the elongation period the speed of the crosshead is constant, similarly as it is done in ASTM D-412 "Rubber Properties in Tension" and other similar tests, the rate of elongation was not held constant. Also, because the elongation period was relatively long, it both influences the peak load and so this may not be a useful value for evaluating the material properties.

The equations for the behavior of crack and joint sealants during the elongation period as well as during

the relaxation period were developed. However, the constitutive equation for the materials could not be formulated, again, because it would require the first half of the test to be performed at the constant elongation rate and thus it would be necessary to precalculate and preprogramme some type of exponential increase of the speed of crosshead in the testing apparatus (if it were technically possible).

Although the test is not designed to produce data in the form best suitable for rheological analysis, it simulates fairly well the process occurring on the road during the cooling period.

REFERENCES

1. R.B. Bird, O. Hassager, R.C. Armstrong, and C.F. Curtiss, Dynamics of Polymeric Liquids, Vol. 1, Wiley, New York, 1977.

TABLE 1. Parameters of Stretch Exponential Model (Crack Sealants from Phase II)

Crack Seal.	a[Pa]	b(-)	c[Pa]	d[-]	λ [s]	A ₁ [s]	A ₂ [s]
2379	0.0351	0.1574	3.9198	0.0941	0.3545	1347.4	592.7
2380	0.0039	0.1442	5.0815	0.0651	0.0052	1582.1	501.1
2381	0.0834	0.2300	2.3494	0.1563	14.4580	962.0	503.1
2382	0.0764	0.1382	3.7546	0.1196	0.7938	1014.2	675.2
2383	0.3149	0.0209	1.0115	0.3128	118.3960	297.9	560.9
2384	0.2122	0.3394	1.2342	0.3694	199.2340	551.7	628.0
2385	0.0948	0.2016	2.3926	0.1715	10.8230	785.5	623.58
2386	0.0477	0.1609	3.6024	0.1265	0.7550	862.4	610.6
2387	0.0635	0.1015	5.5165	0.1049	0.0371	858.5	697.4
2388	0.3243	0.2944	1.0290	0.2913	475.9480	801.7	670.1
2389	0.0796	0.1877	2.7142	0.1188	5.4570	1192.4	616.6
2390	0.1267	0.2568	1.7628	0.2418	45.1300	619.0	606.4
2448	0.3384	0.0470	0.8705	0.3402	414.0830	485.7	615.2
2449	0.0454	0.1117	5.0767	0.0889	0.0304	1150.3	673.7
2450	0.0144	0.1039	6.2457	0.0650	0.000666	1416.9	635.1
2451	0.0340	0.5441	1.3116	0.2359	607.1020	1205.6	628.8

TABLE 2. Parameters of Stretch Exponential Model (Crack Sealants from Phase IV)

Crack Sealant		a [Pa]	b(-)	c[Pa]	d[-]	λ [s]	A ₁ [s]	A ₂ [s]
Sample 1	(1)	0.280	0.408	1.148	0.243	429.0	947.3	529.7
	(2)	0.244	0.487	1.200	0.225	371.7	971.9	499.8
Sample 2	(2)	0.314	0.404	1.318	0.202	512.9	1186.8	552.7
	(3)	0.315	0.369	1.327	0.194	493.8	1193.0	566.1
	(4)	0.256	0.311	1.428	0.190	270.7	1129.3	565.3
Sample 4	(2)	0.211	0.534	1.269	0.241	314.0	957.5	475.4
	(3)	0.229	0.527	1.282	0.225	345.7	1018.8	487.3
Sample 5	(1)	0.305	0.424	1.369	0.196	475.4	1217.4	551.5
	(2)	0.292	0.451	1.411	0.180	455.0	1263.1	536.8
Sample 6	(1)	0.320	0.202	1.127	0.235	422.1	938.1	614.6
	(2)	0.288	0.328	1.158	0.259	398.0	916.8	558.5
	(3)	0.256	0.184	1.463	0.181	150.9	1021.5	603.5
Sample 10	(1)	0.309	0.330	1.130	0.250	508.3	972.5	556.3
	(2)	0.312	0.341	1.136	0.252	520.3	983.0	556.3
Sample 11	(1)	0.0787	0.086	5.729	0.096	0.0141	887.2	624.5
	(2)	0.0706	0.0758	7.159	0.0865	0.00222	941.6	615.72
	(3)	0.326	0.154	1.140	0.238	378.3	916.2	637.4
Sample 12	(1)	0.312	0.459	1.478	0.176	515.6	1361.1	544.0
	(2)	0.323	0.445	1.455	0.174	545.3	1356.3	547.3
Sample 14	(1)	0.198	0.496	1.496	0.186	270.6	1188.0	473.9
	(2)	0.325	0.426	1.317	0.212	520.8	1175.9	561.3

**TABLE 3. Stress Relaxation Test
Area Under Relaxation (Normalized)**

Crack Sealant	A₁[s]	Sample	A₁[s]
2383	297.9	4(3)	1018.8
2448	485.7	2(4)	1129.3
2384	551.7	2449	1150.3
2390	619.0	14(2)	1175.9
2385	785.5	2(2)	1186.8
2388	801.8	14(1)	1188.0
2387	858.5	2389	1192.4
2386	862.4	2(3)	1193.0
11(3)	916.2	2451	1205.6
6(2)	916.8	5(1)	1217.4
6(1)	938.1	5(2)	1263.1
11(2)	941.6	2379	1347.4
1(1)	947.3	12(2)	1356.3
4(2)	957.5	12(1)	1361.1
2381	961.9	2380	1582.1
1(2)	971.9	2450	1416.9
10(1)	972.5		
10(2)	983.0		
2382	1014.2		

FIGURE 1. Reduced Extra-Stress Component, Sample 2383

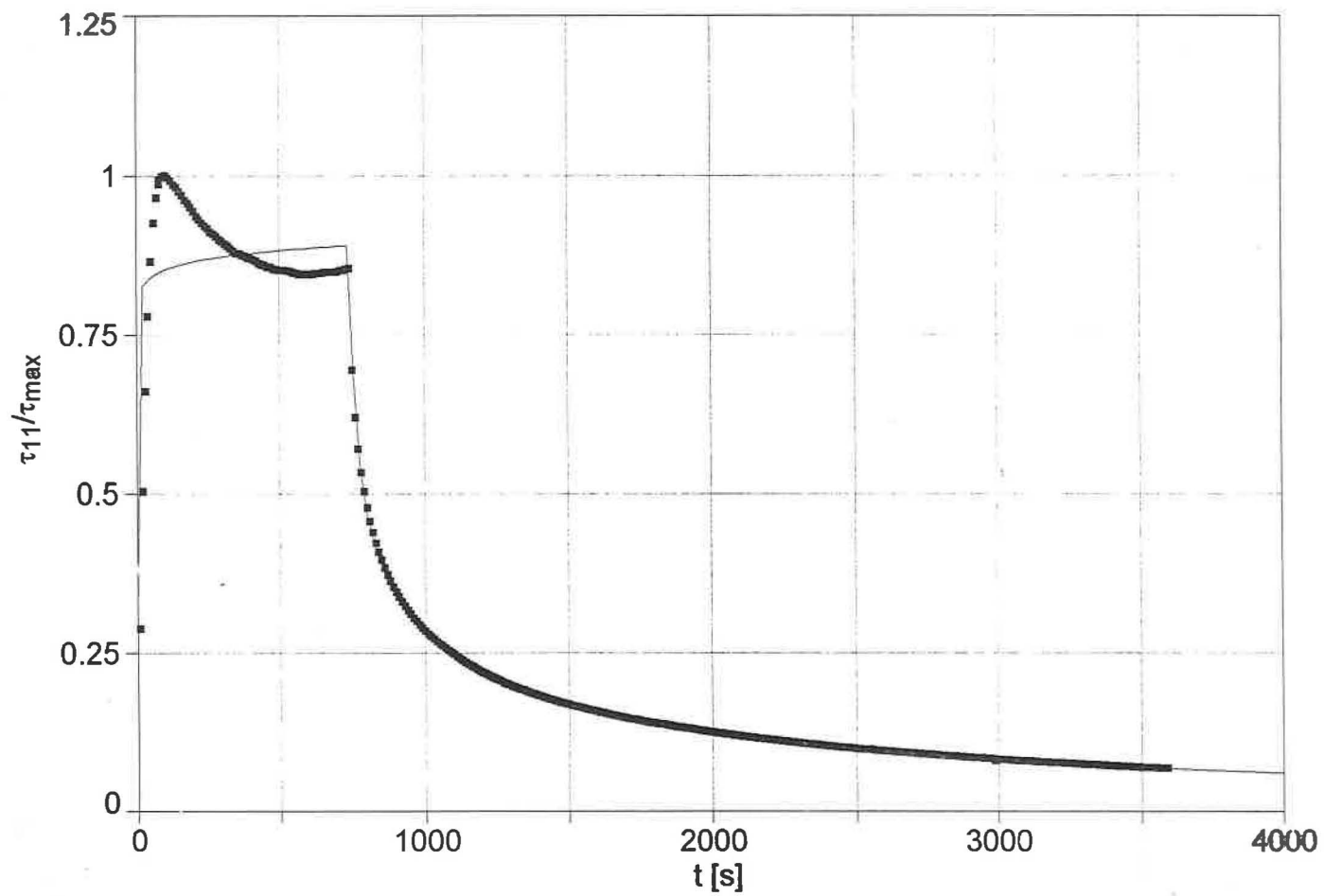


FIGURE 2. Reduced Extra-Stress Component, Sample 2448

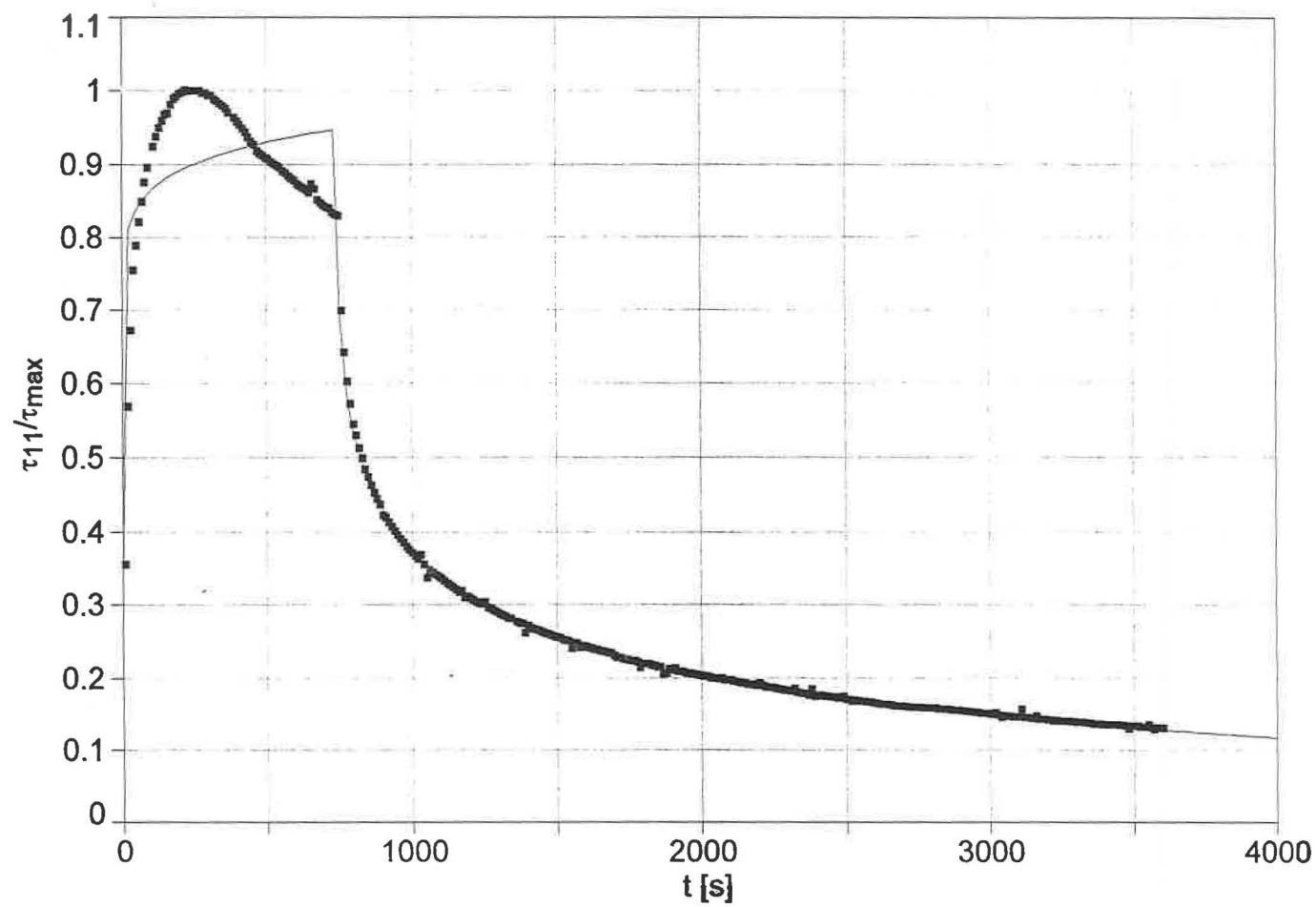


FIGURE 3. Reduced Extra-Stress Component, Sample 2384

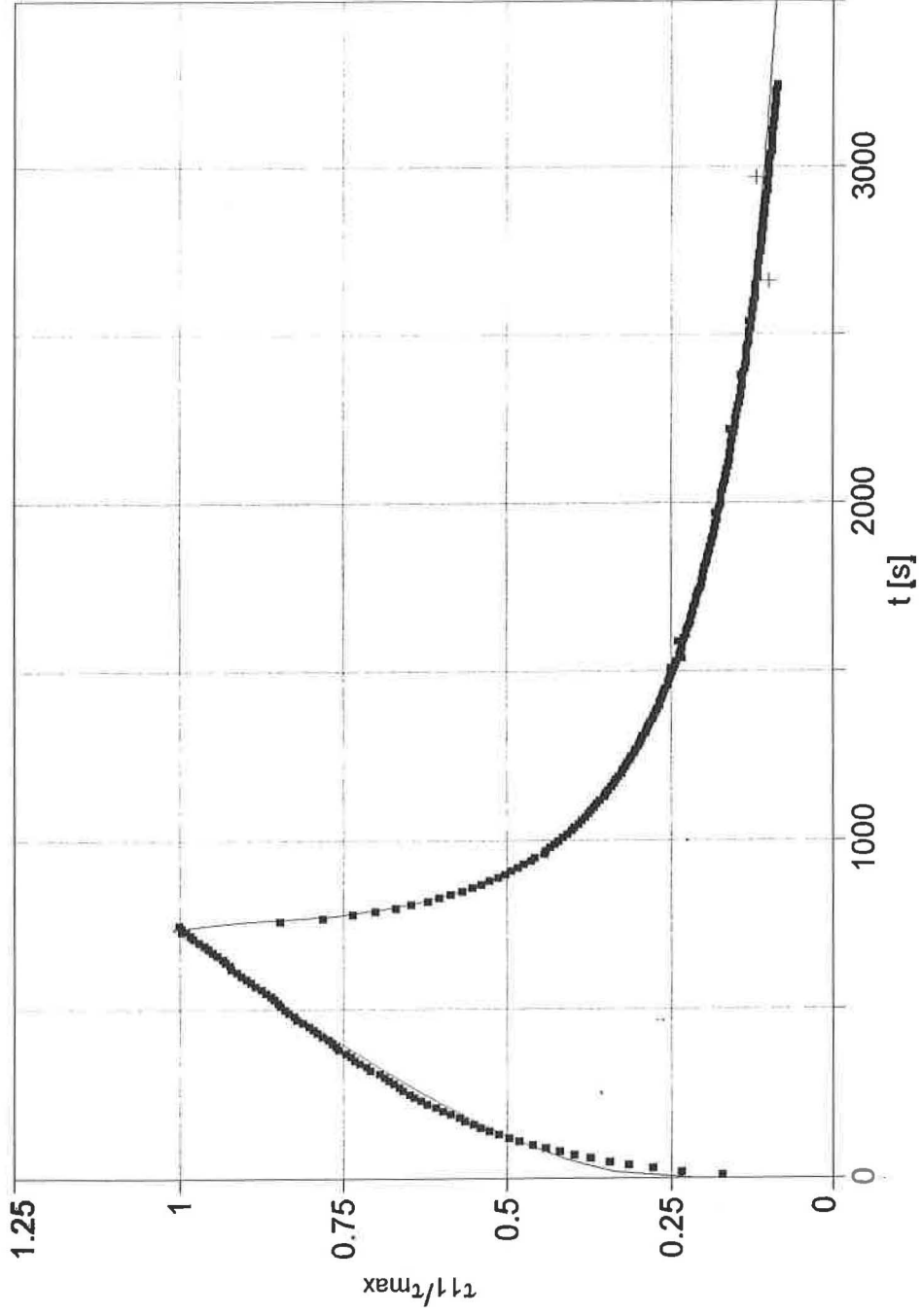


FIGURE 4. Reduced Extra-Stress Component, Sample 2390

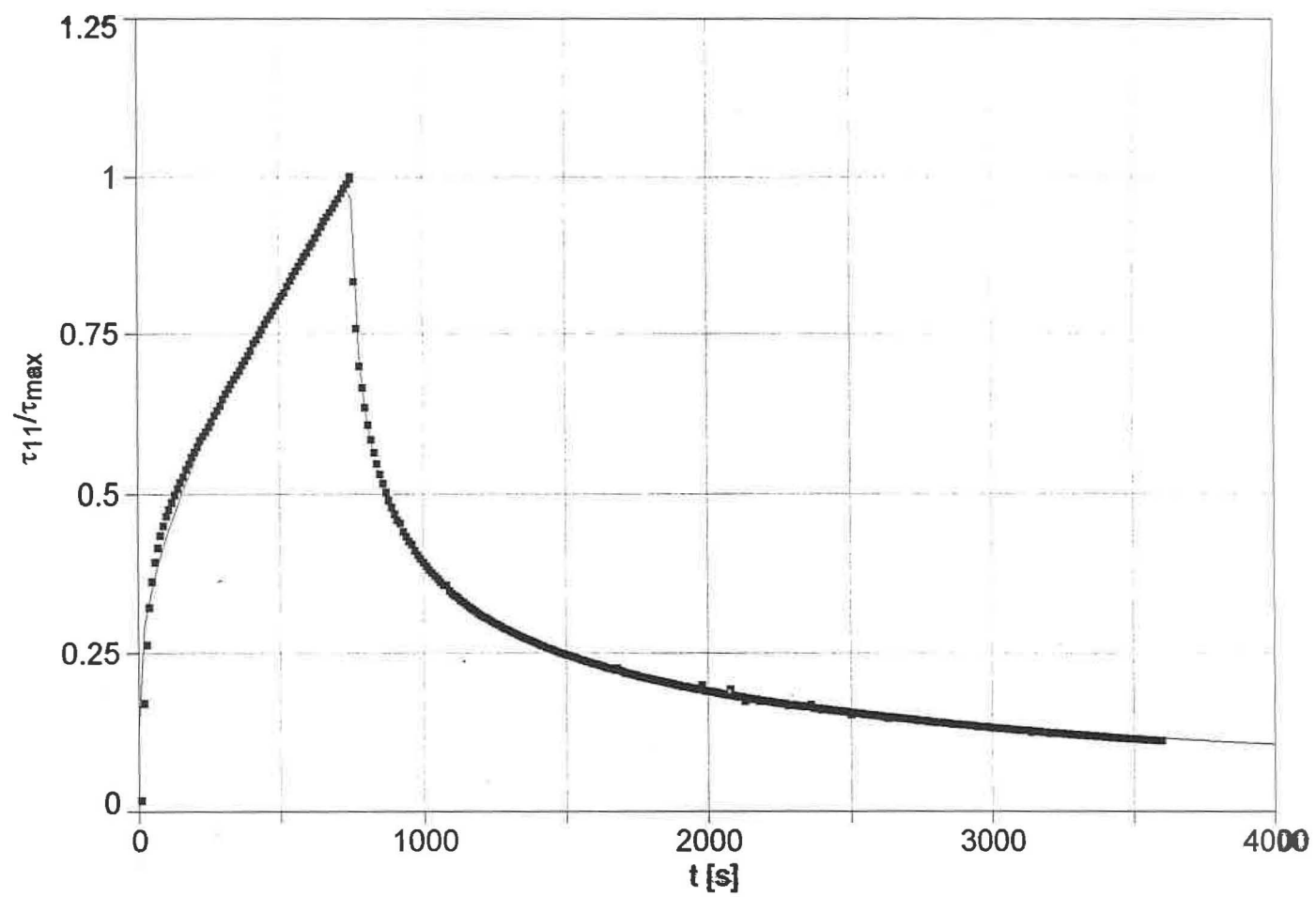


FIGURE 5. Reduced Extra-Stress Component, Sample 2385

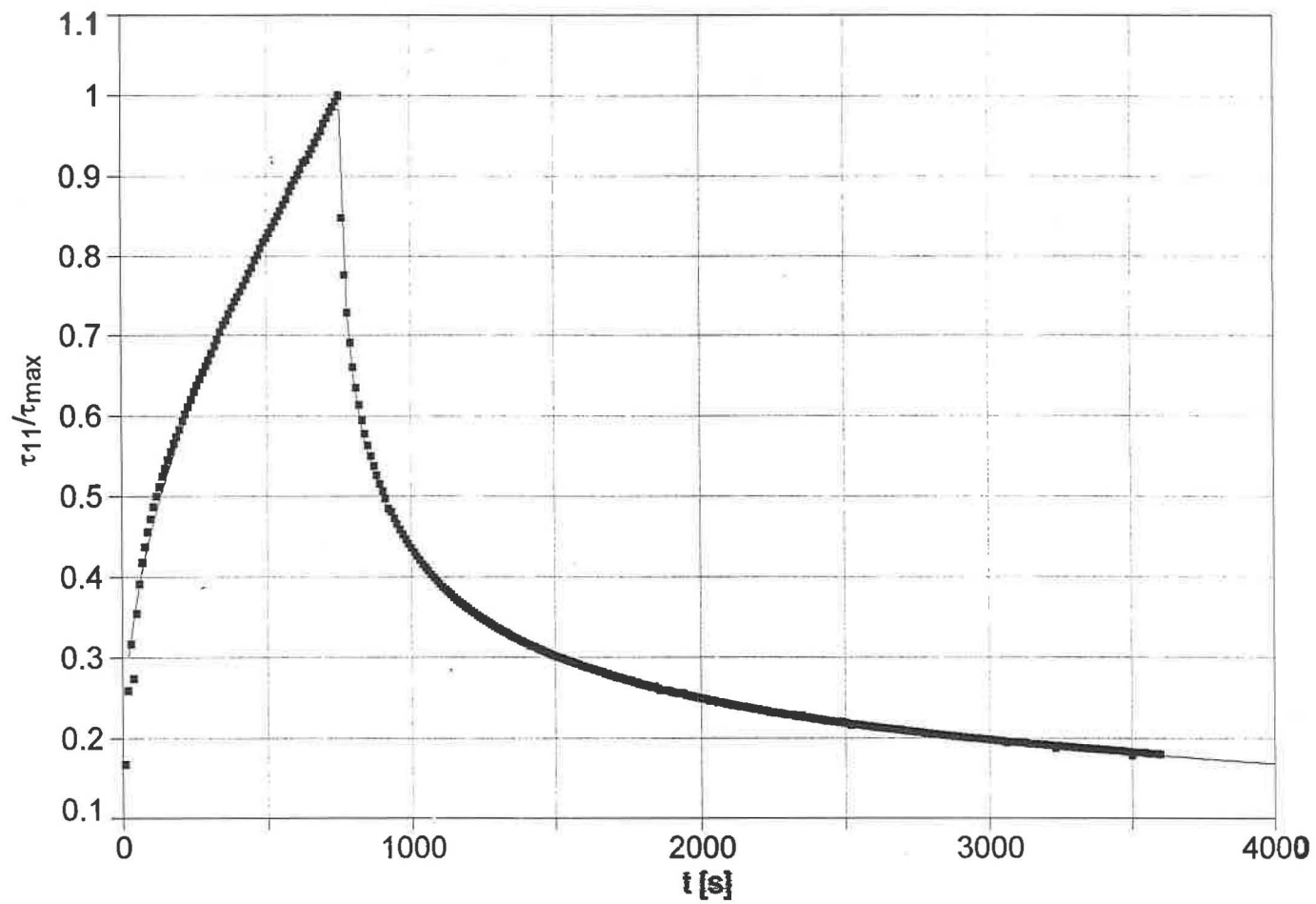


FIGURE 6. Reduced Extra-Stress Component, Sample 2388

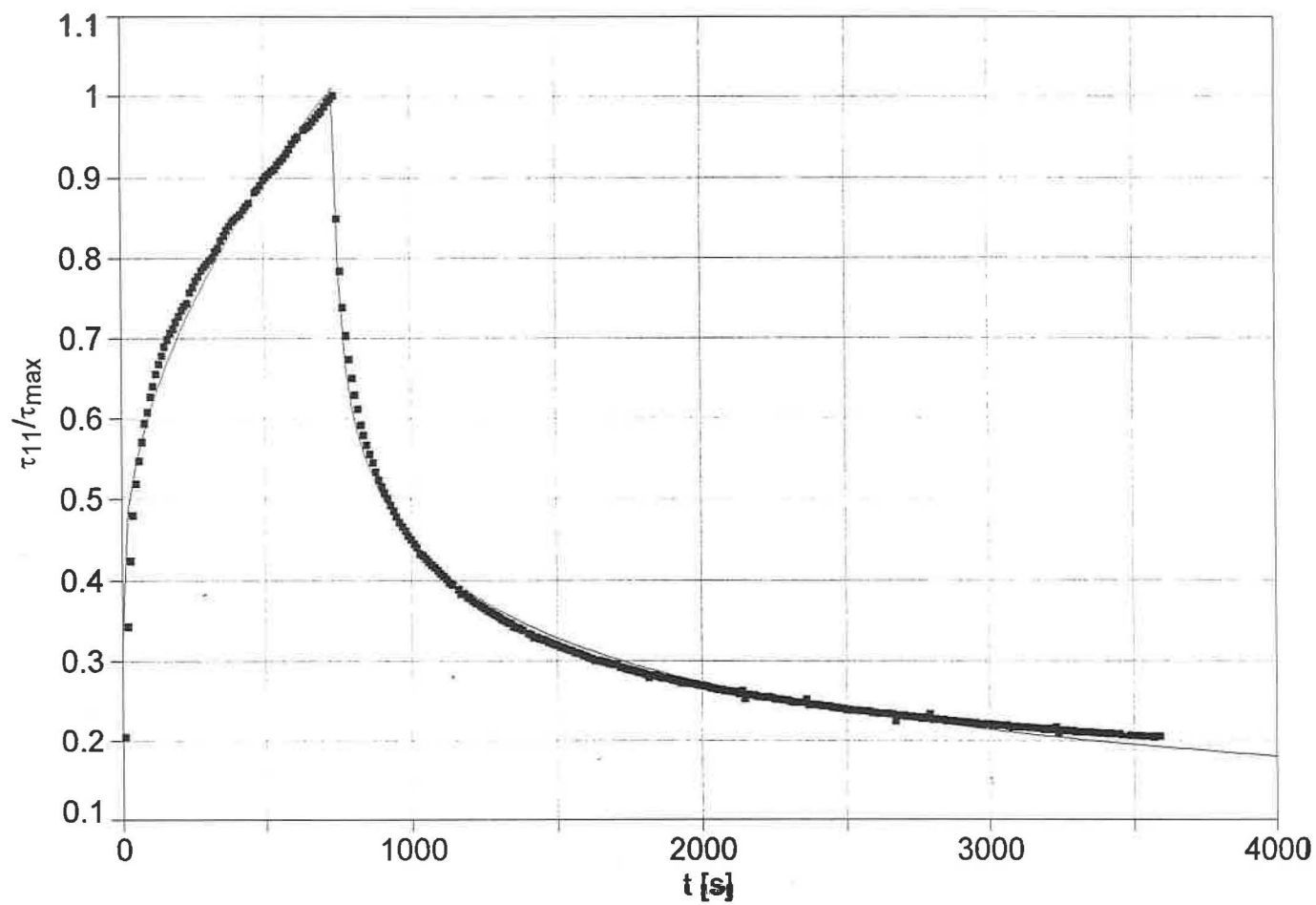


FIGURE 7. Reduced Extra-Stress Component, Sample 2387

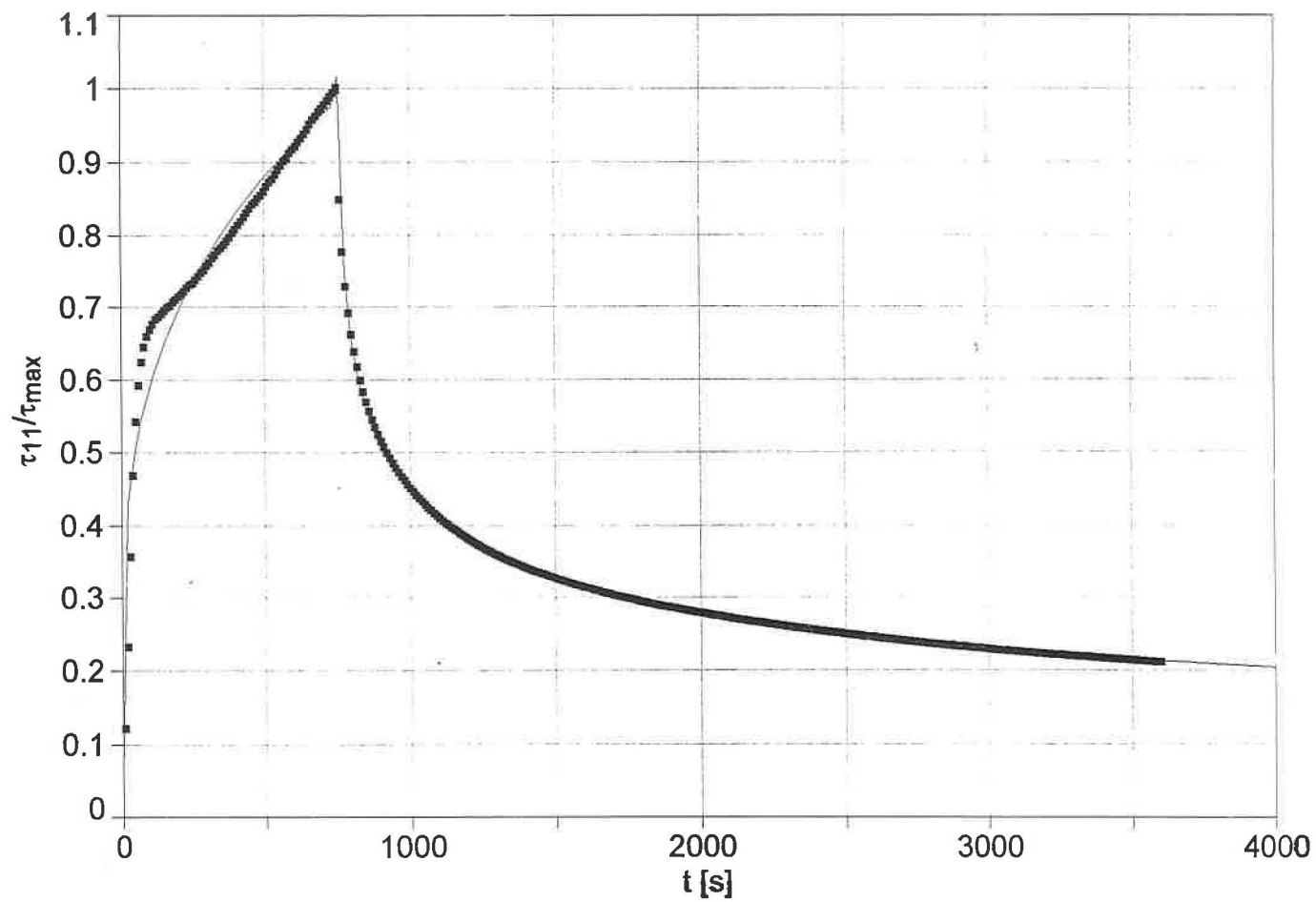


FIGURE 8. Reduced Extra-Stress Component, Sample 2386

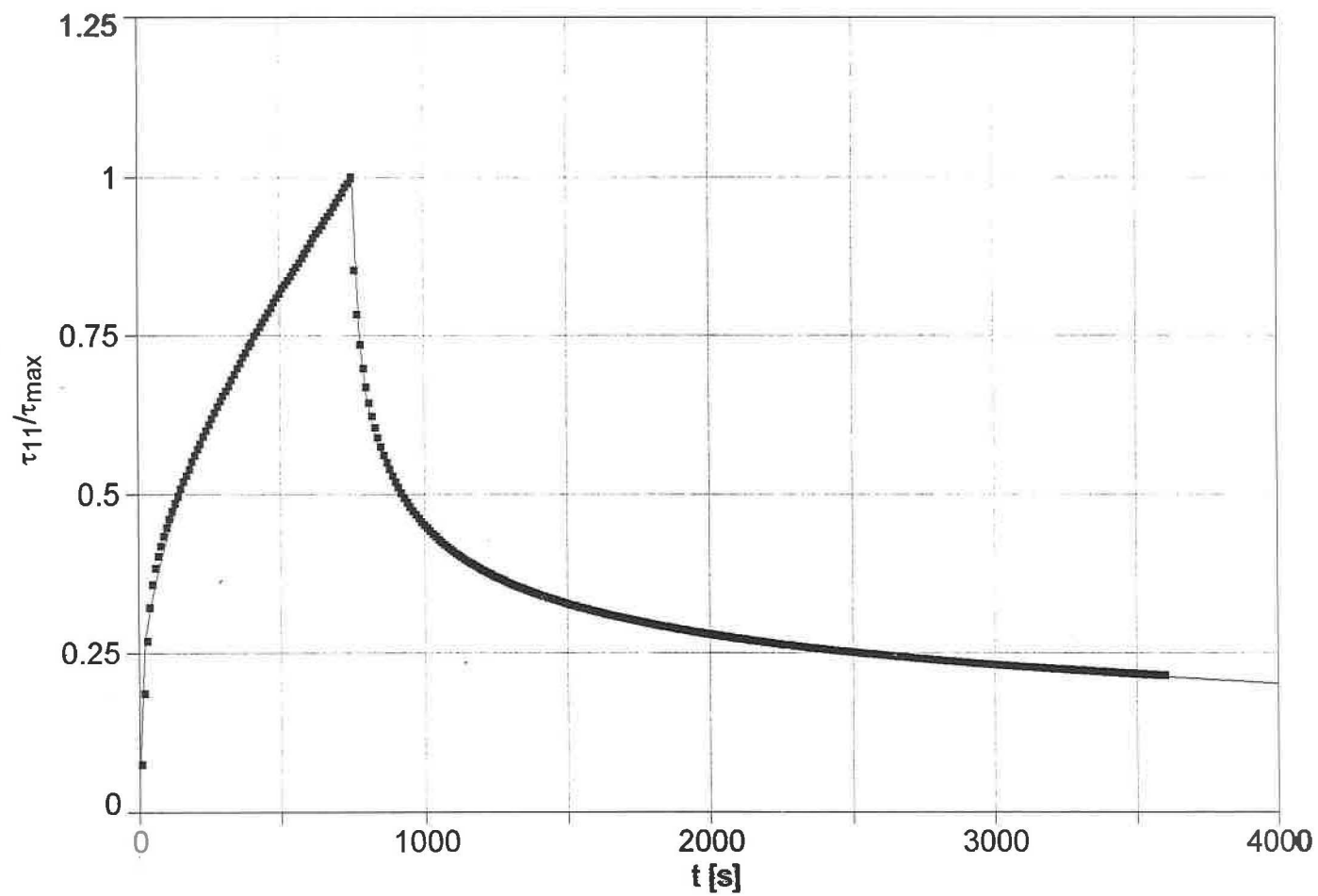


FIGURE 9. Reduced Extra-Stress Component, Sample 11(1)

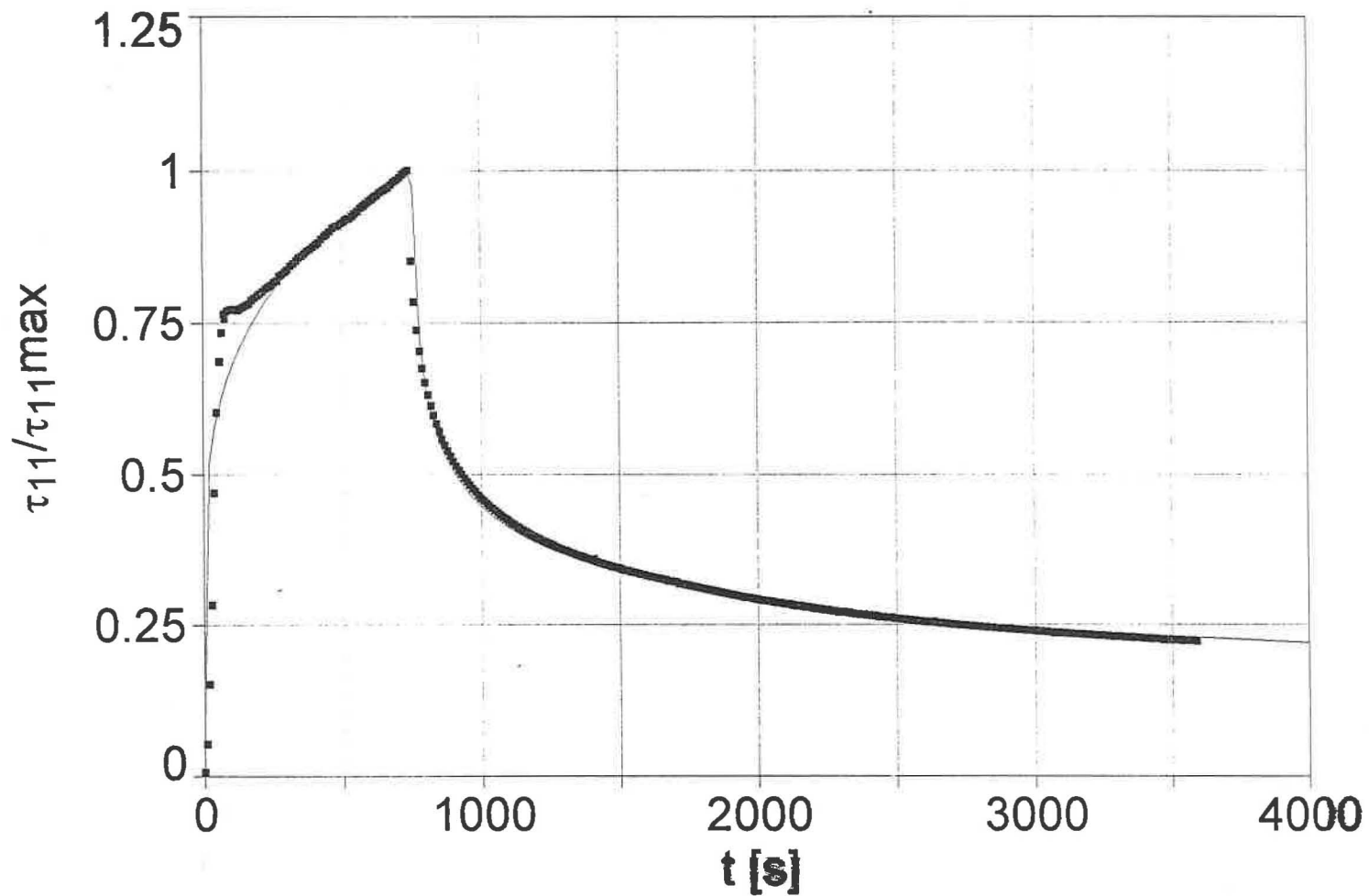


FIGURE 10. Reduced Extra-Stress Component, Sample 11(3)

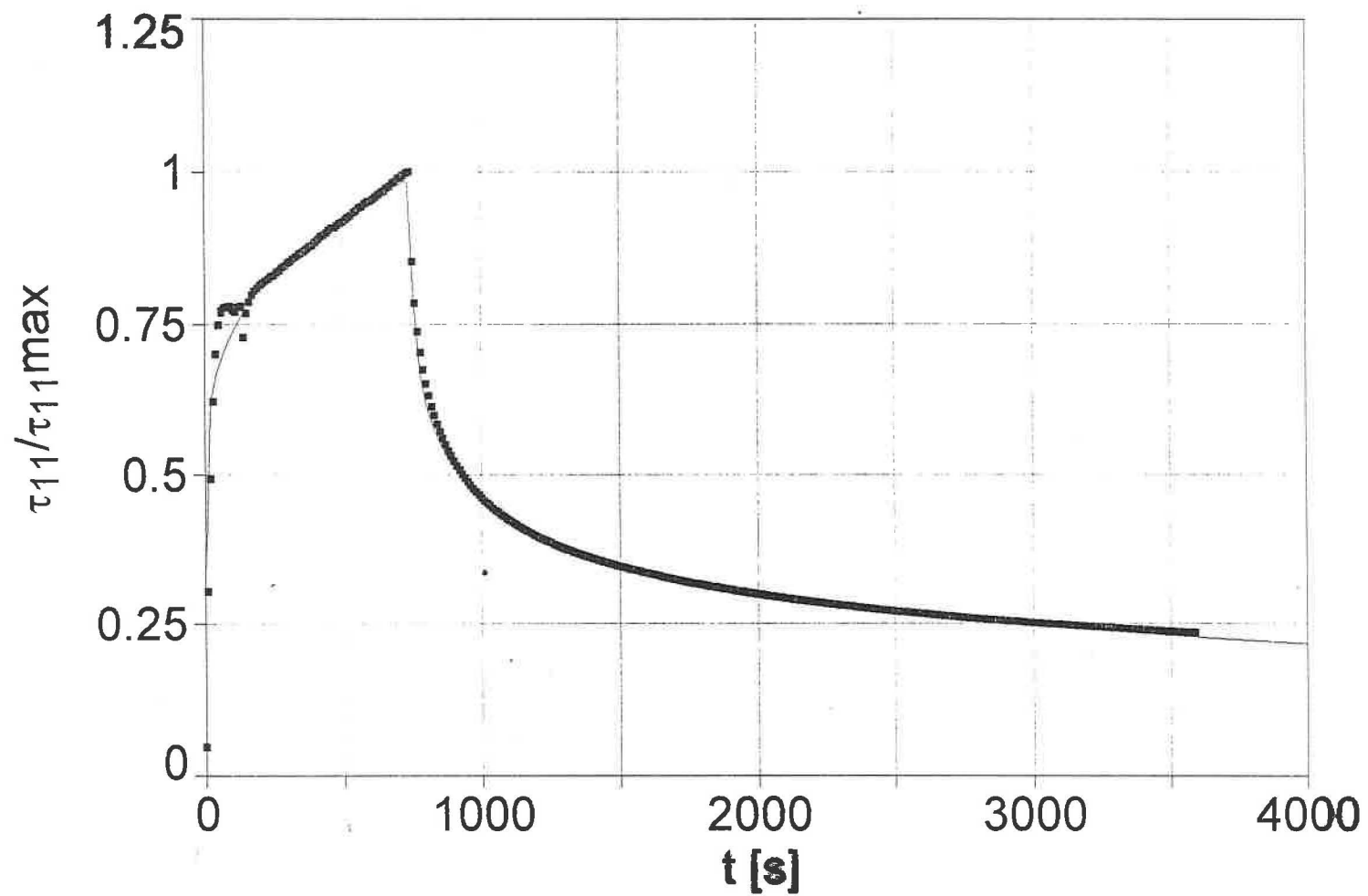


FIGURE 11. Reduced Extra-Stress Component, Sample 6(2)

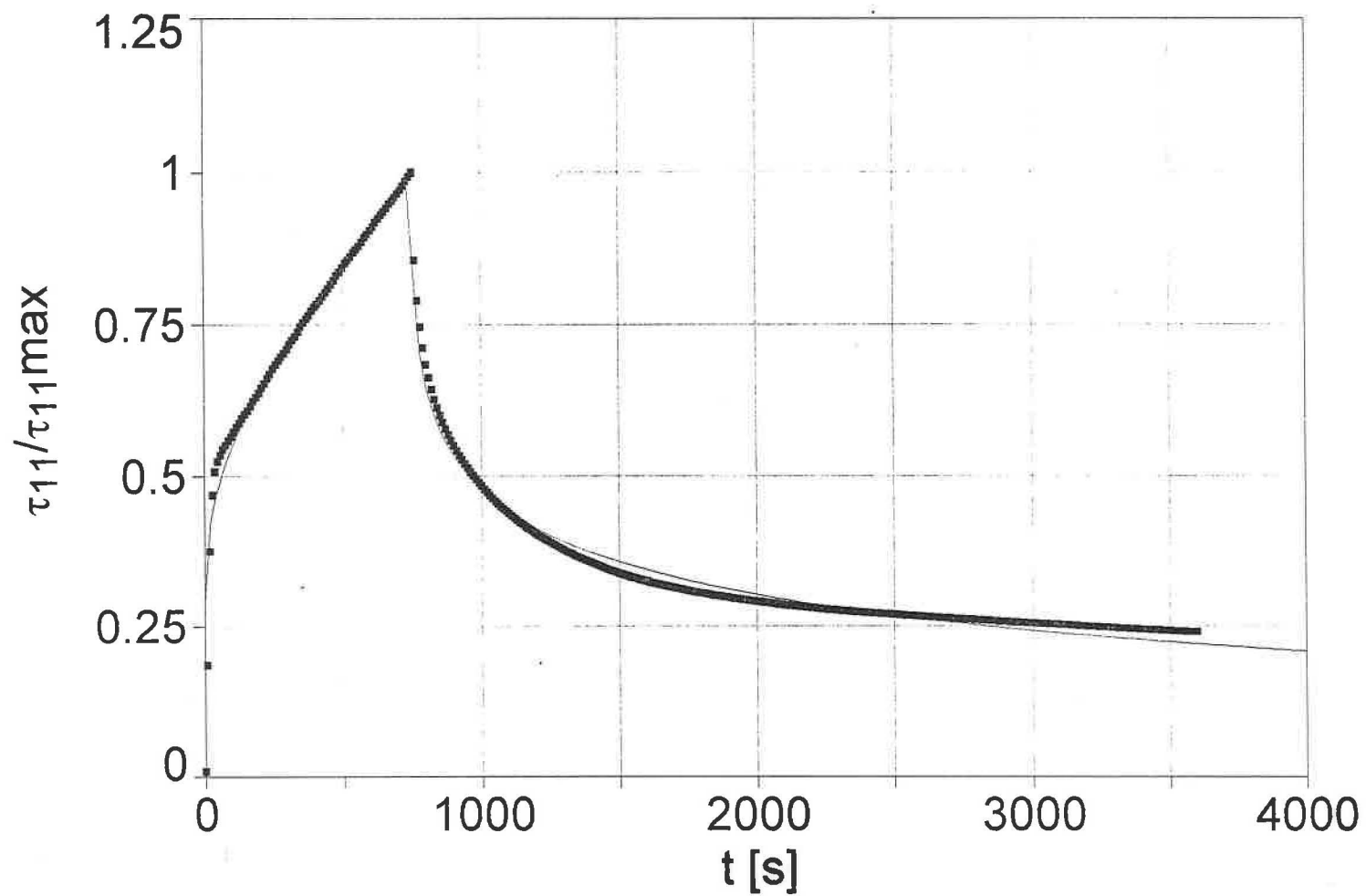


FIGURE 12. Reduced Extra-Stress Component, Sample 6(1)

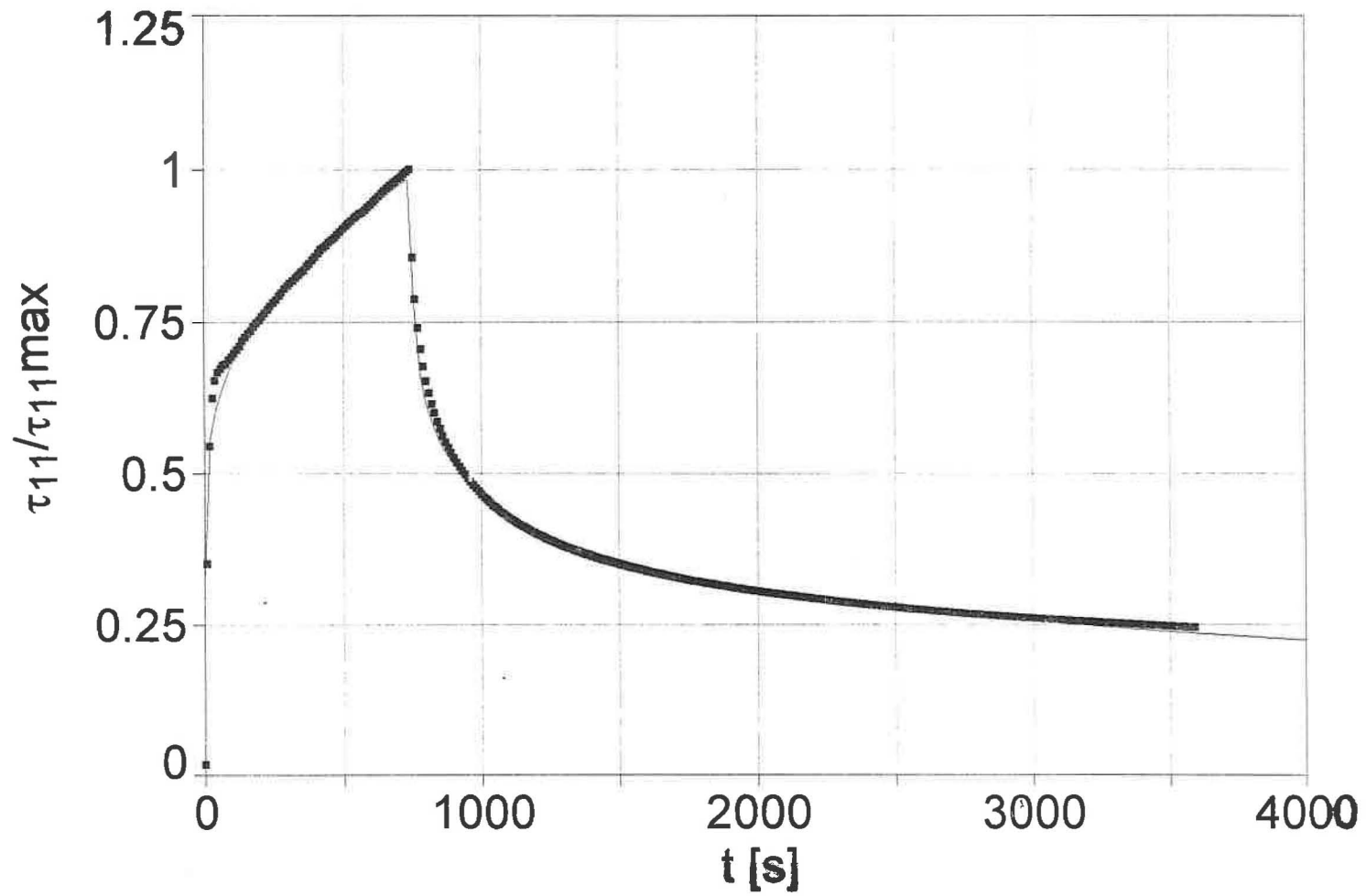


FIGURE 13. Reduced Extra-Stress Component, Sample 11(2)

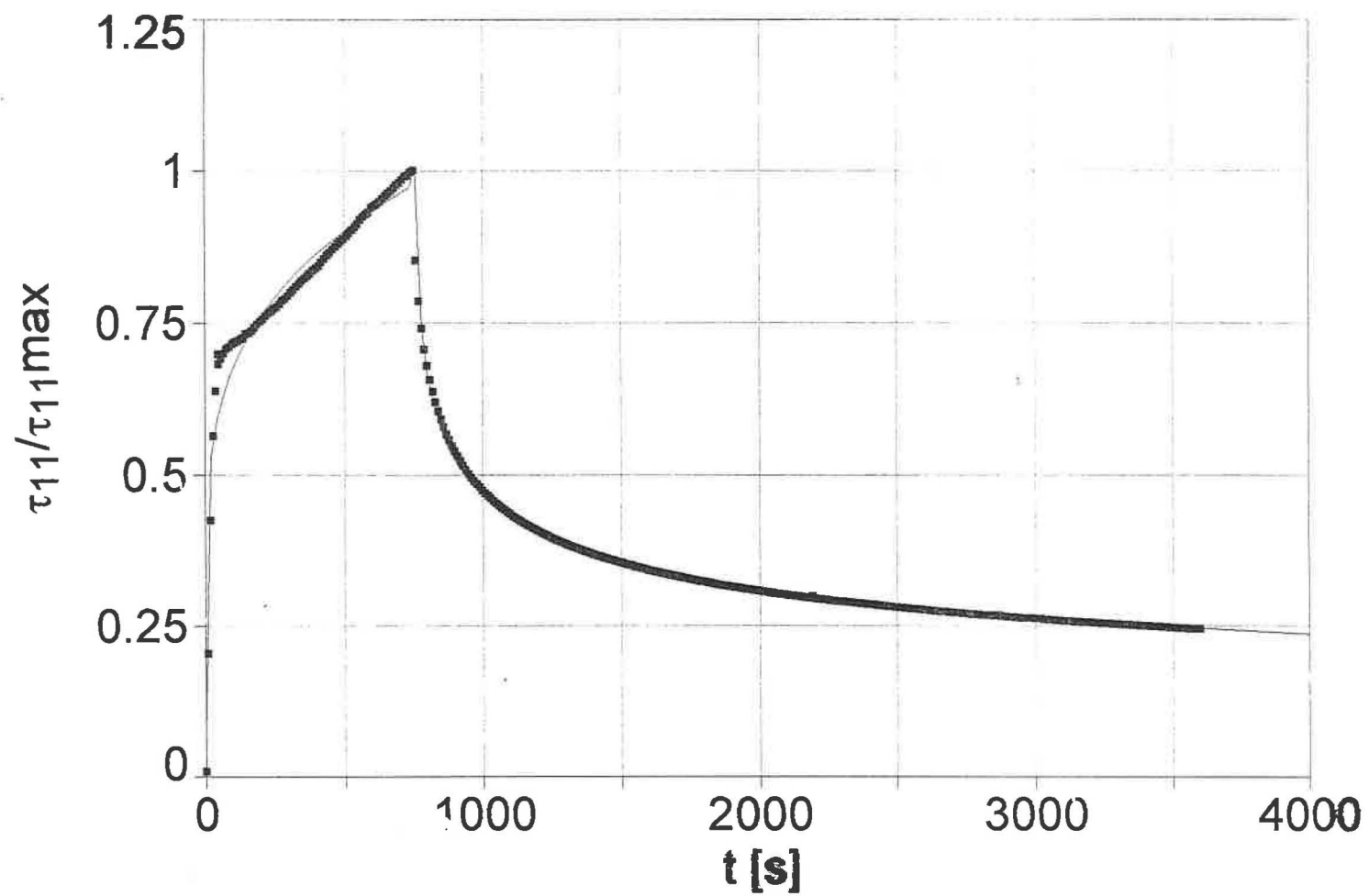


FIGURE 14. Reduced Extra-Stress Component, Sample 1(1)

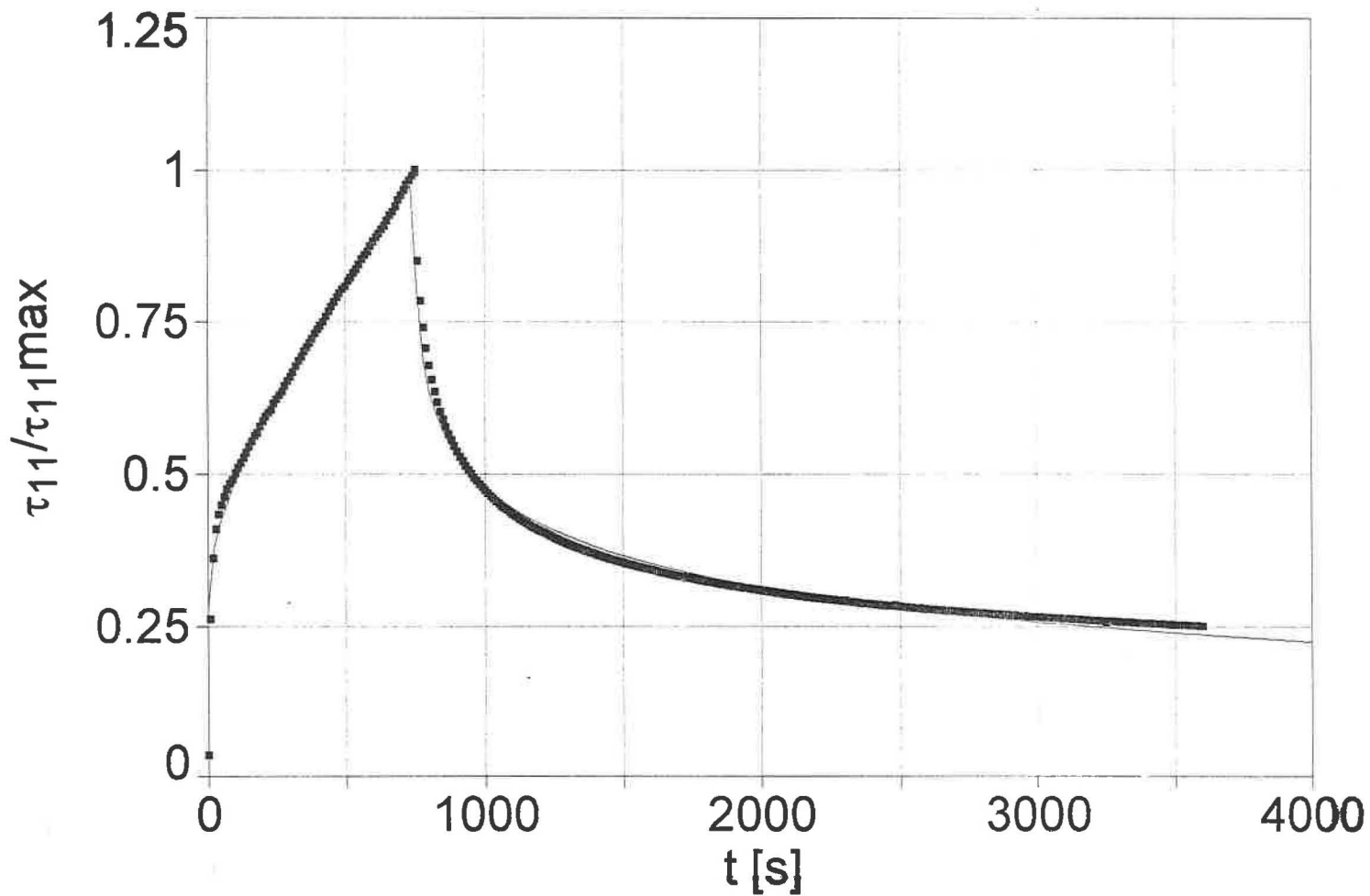


FIGURE 15. Reduced Extra-Stress Component, Sample 4(2)

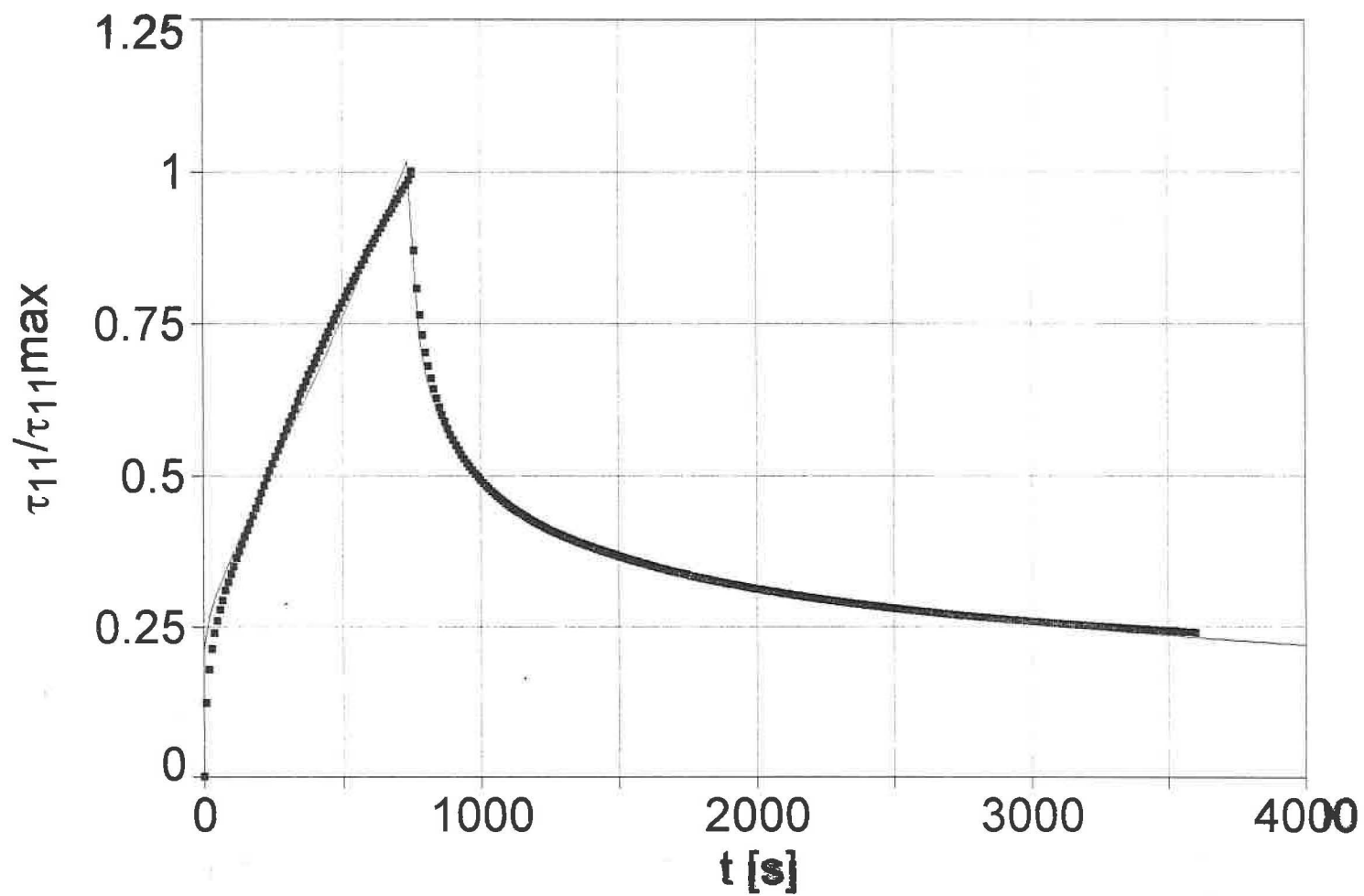


FIGURE 16. Reduced Extra-Stress Component, Sample 2381

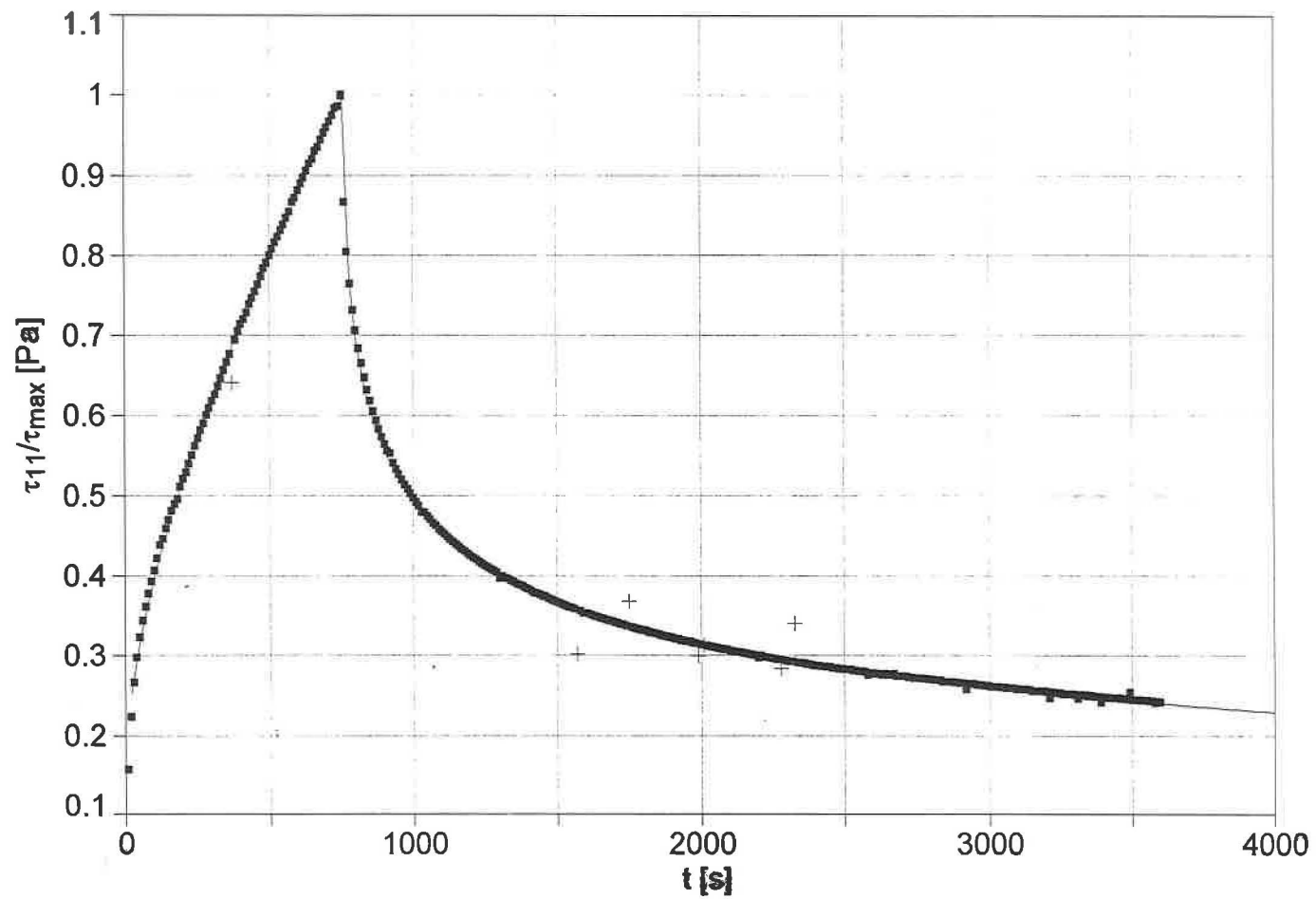


FIGURE 17. Reduced Extra-Stress Component, Sample 1(2)

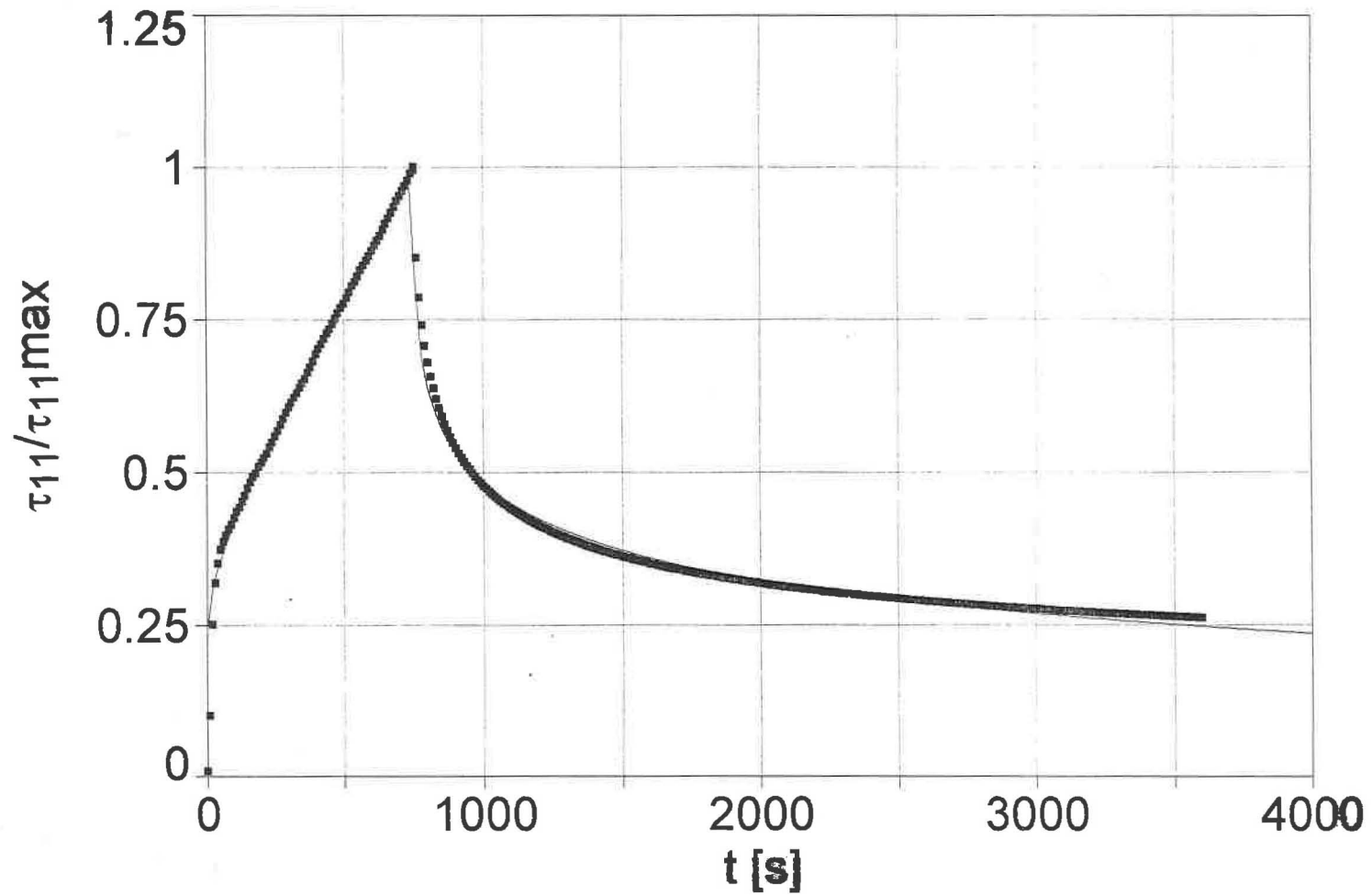


FIGURE 18. Reduced Extra-Stress Component, Sample 10(1)

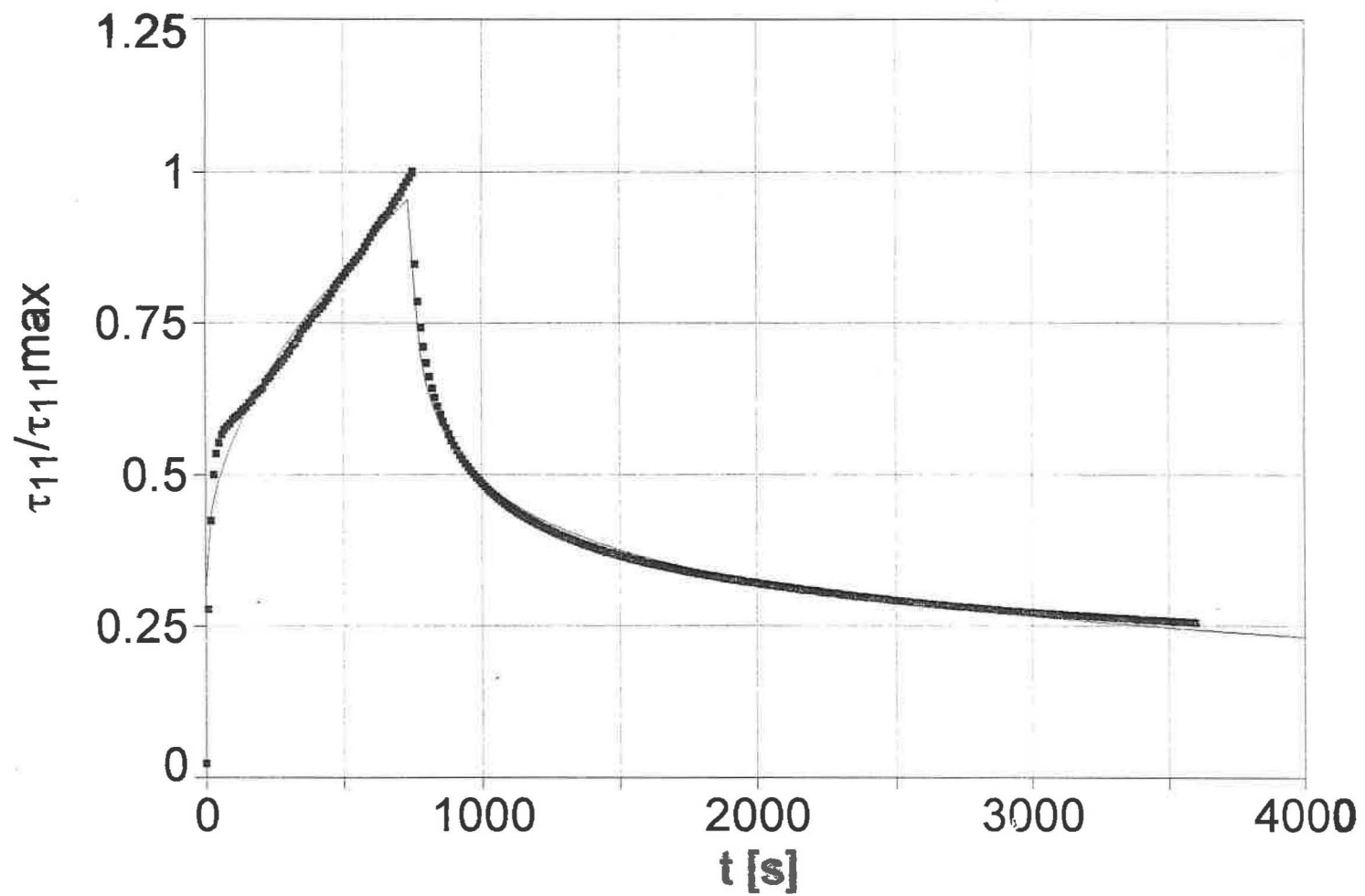


FIGURE 19. Reduced Extra-Stress Component, Sample 10(2)

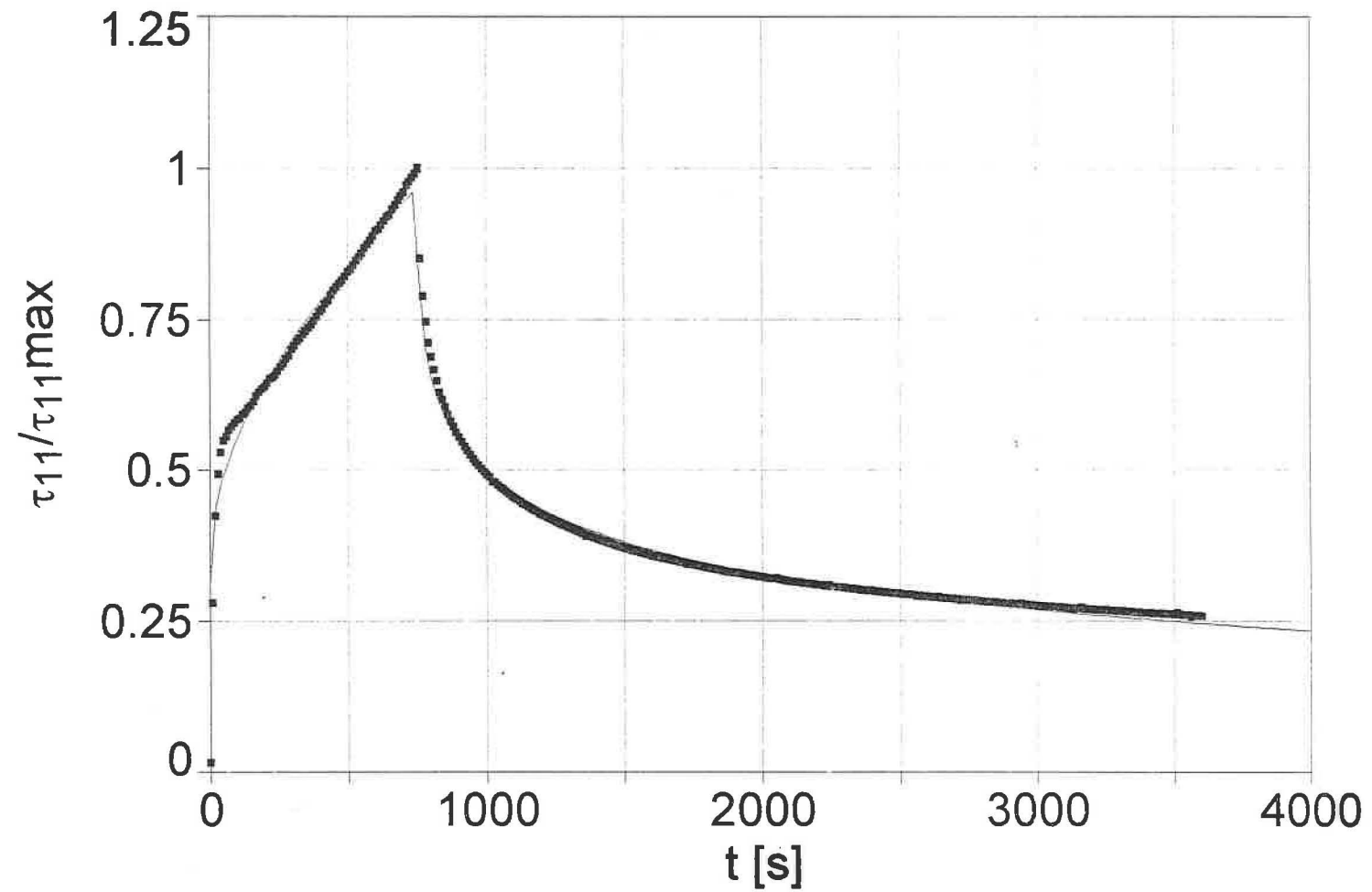


FIGURE 20. Reduced Extra-Stress Component, Sample 2382

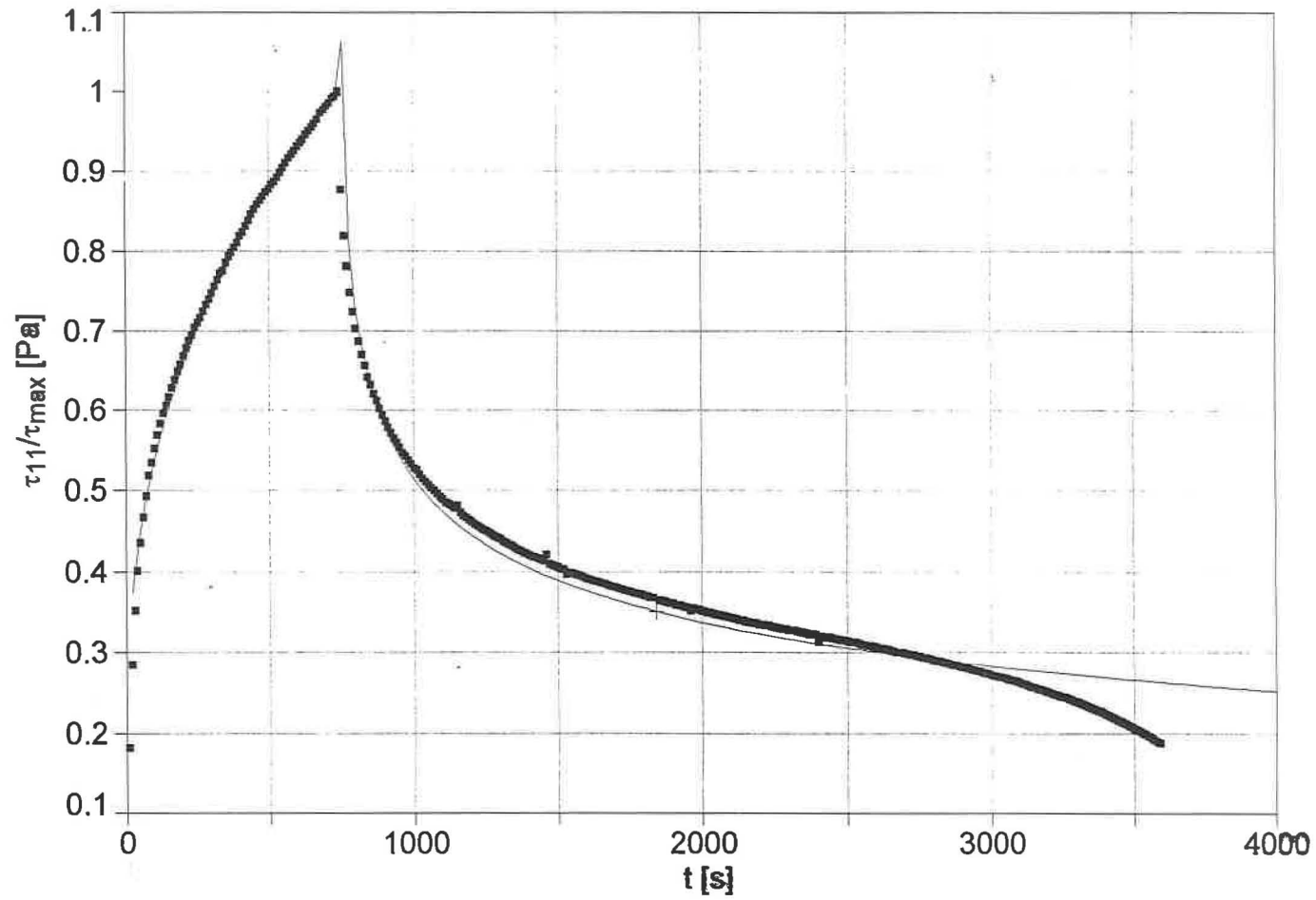


FIGURE 21. Reduced Extra-Stress Component, Sample 4(3)

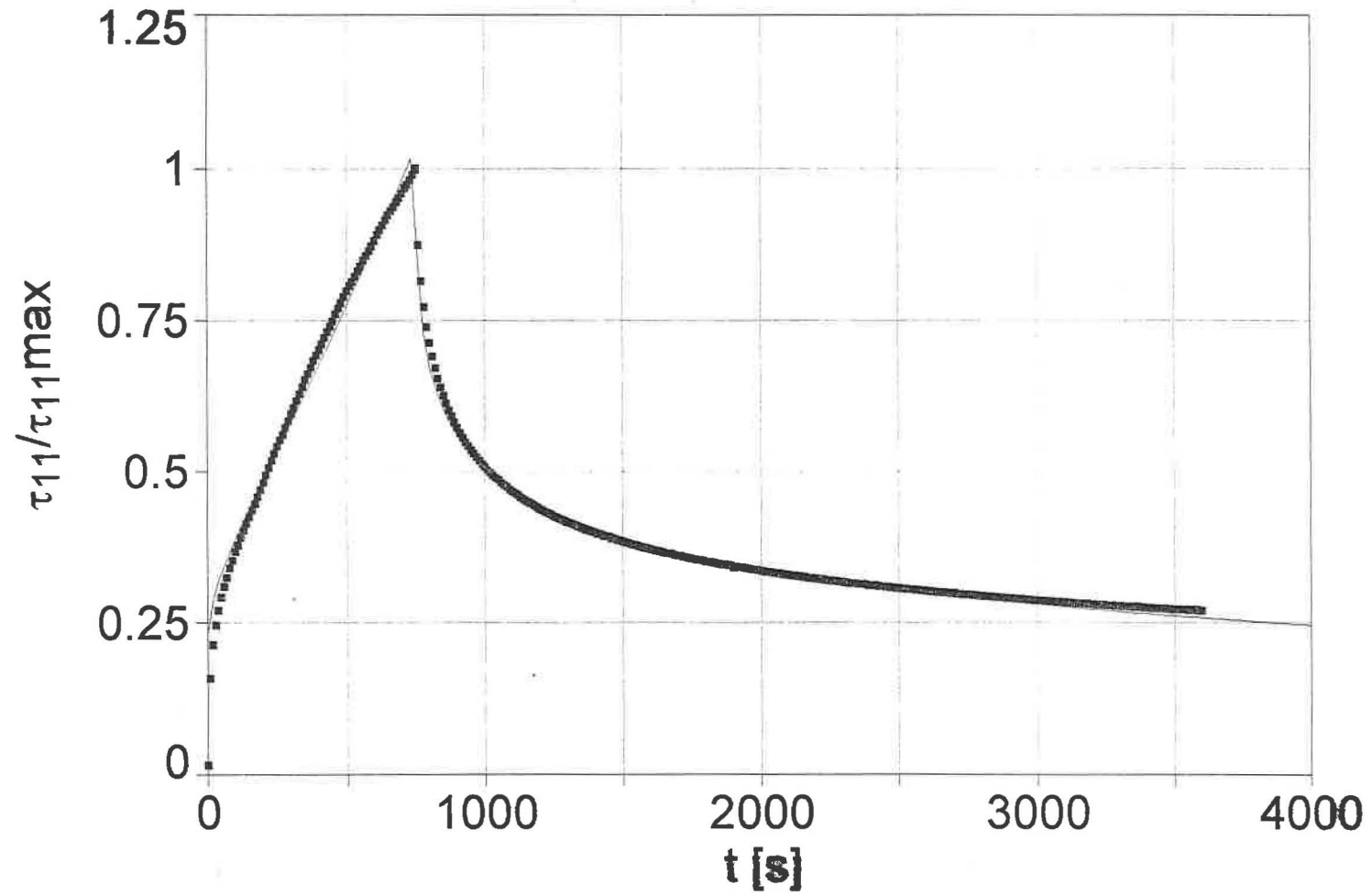


FIGURE 22. Reduced Extra-Stress Component, Sample 6(3)

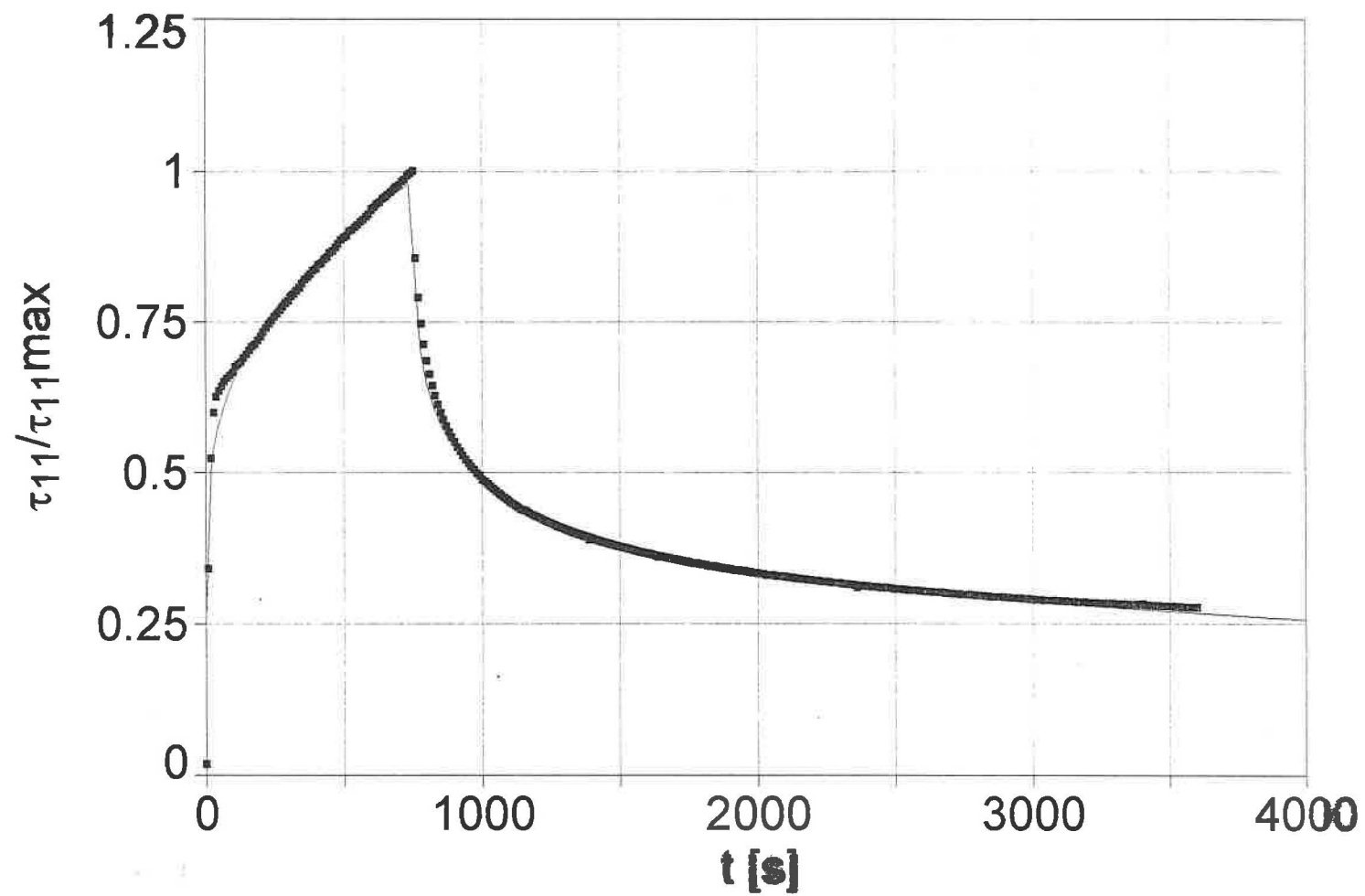


FIGURE 23. Reduced Extra-Stress Component, Sample 2(4)

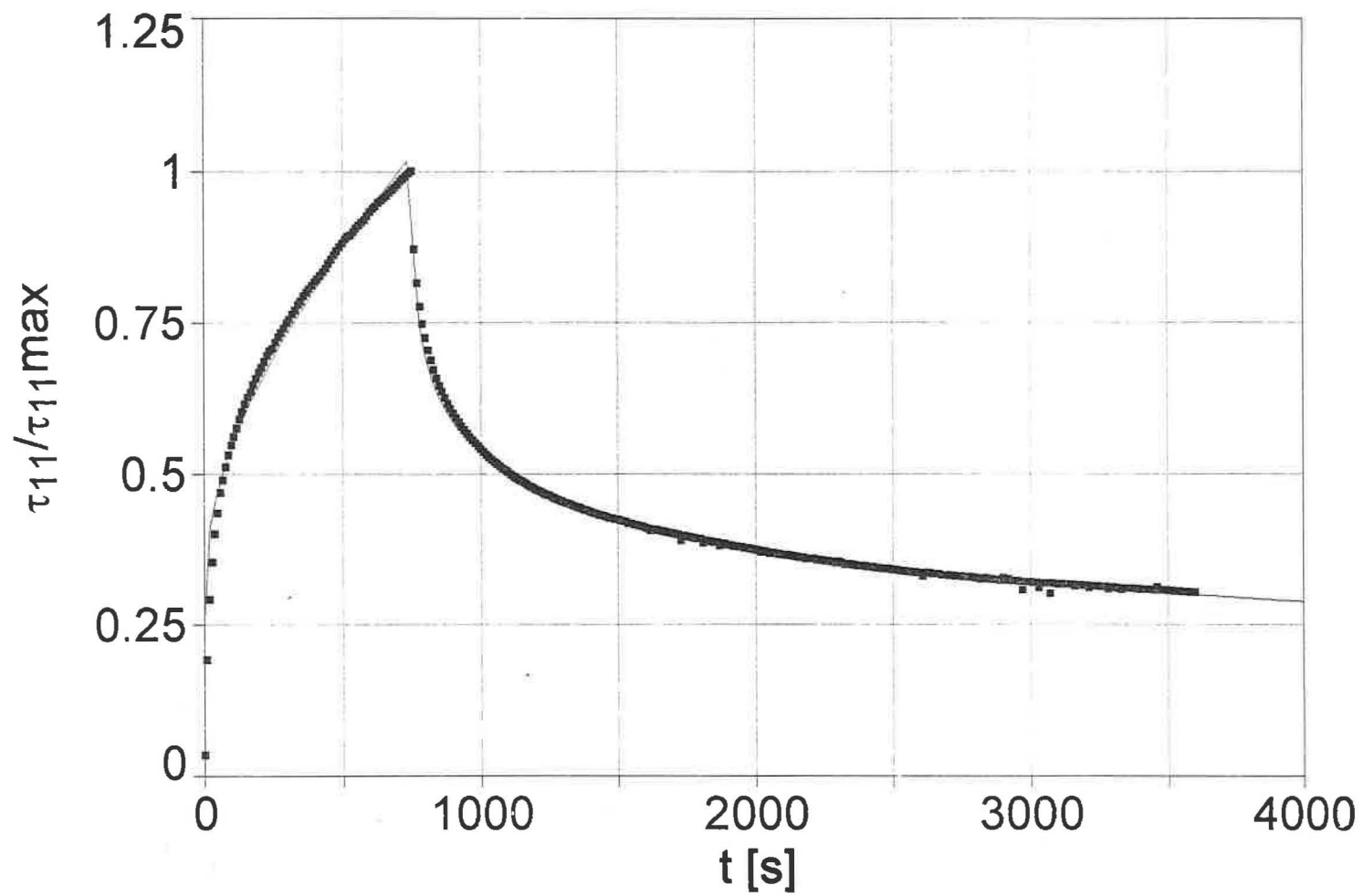


FIGURE 24. Reduced Extra-Stress Component, Sample 2449

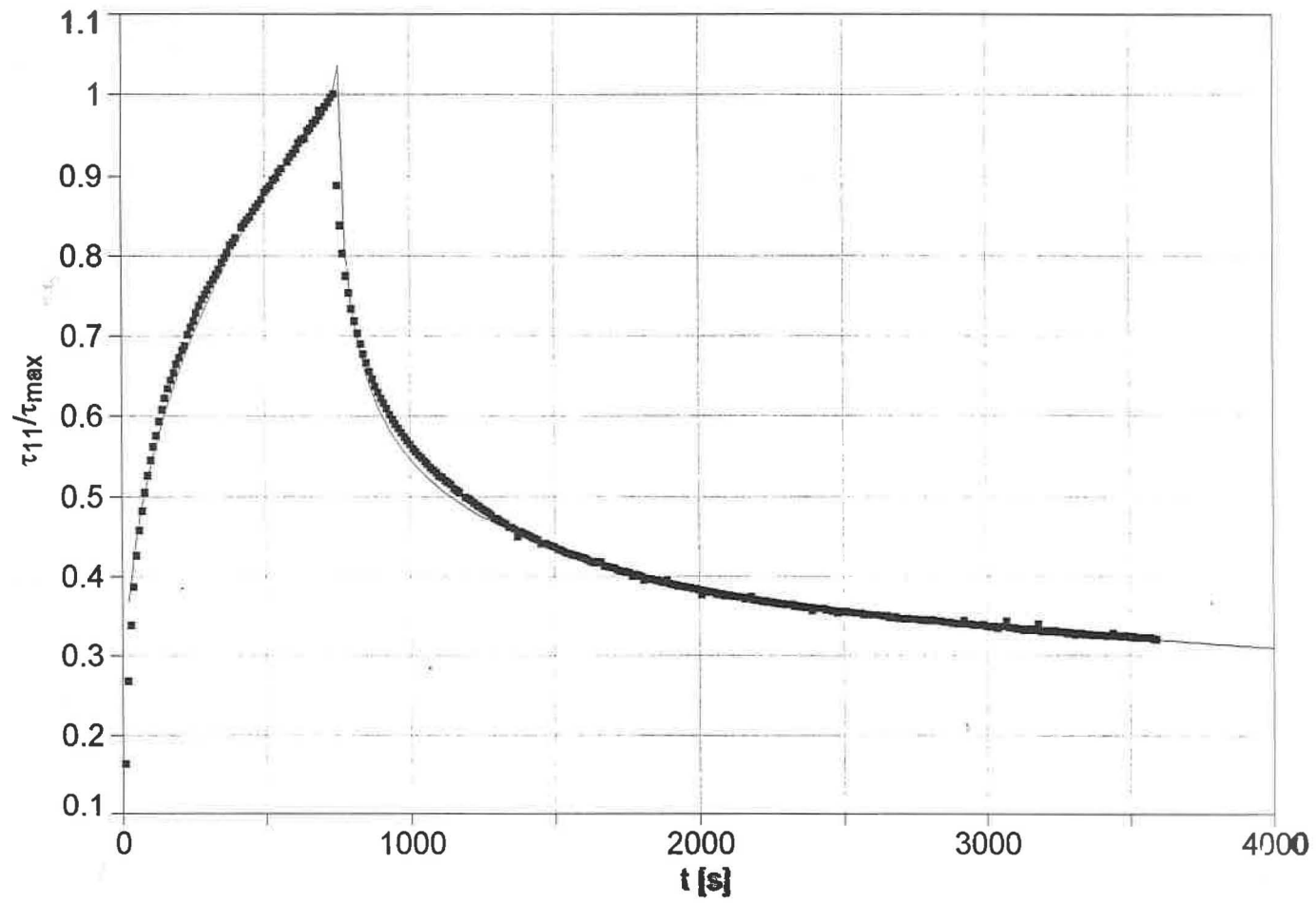


FIGURE 25. Reduced Extra-Stress Component, Sample 14(1)

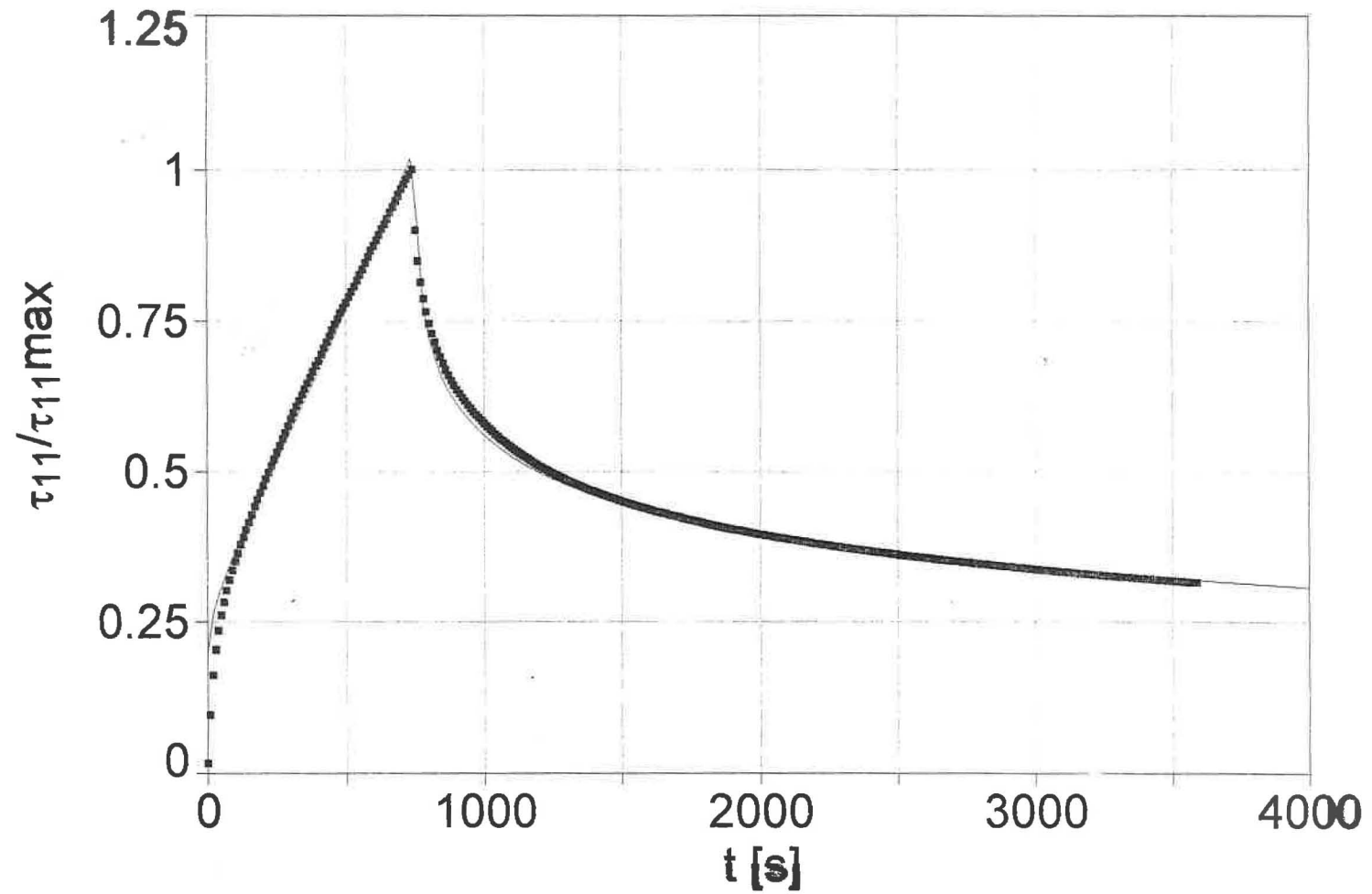


FIGURE 26. Reduced Extra-Stress Component, Sample 2389

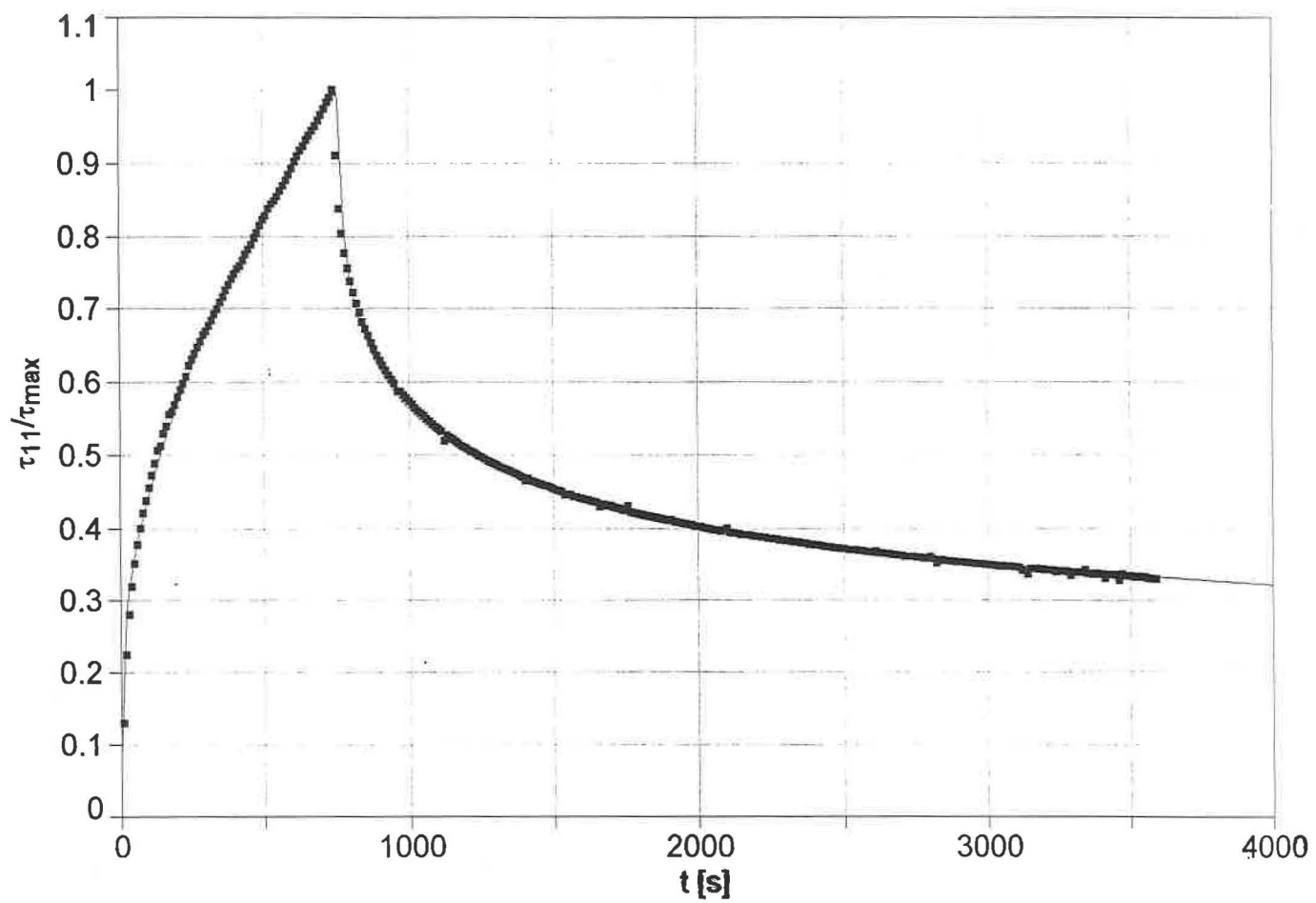


FIGURE 27. Reduced Extra-Stress Component, Sample 2(3)

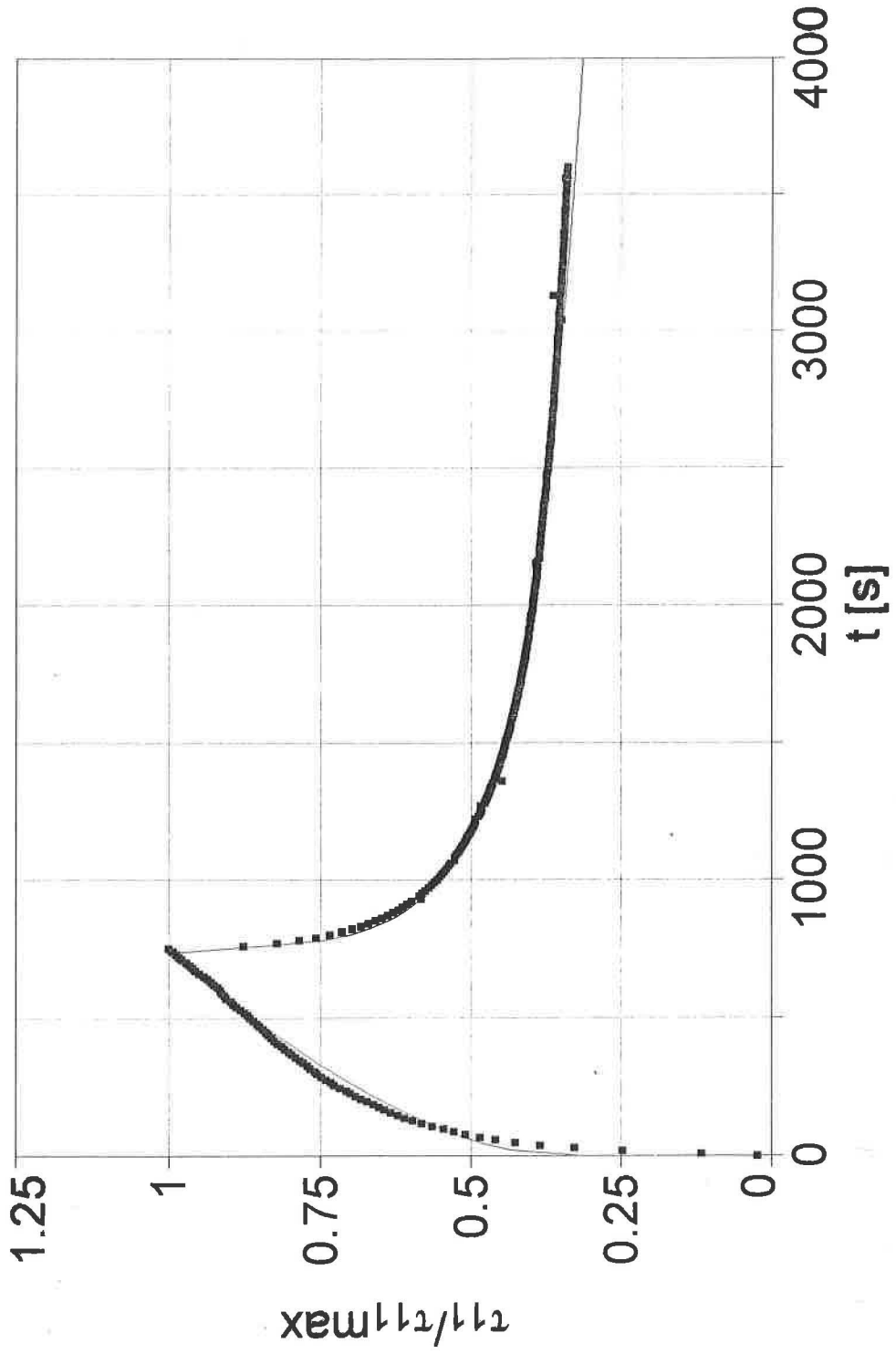


FIGURE 28. Reduced Extra-Stress Component, Sample 2451

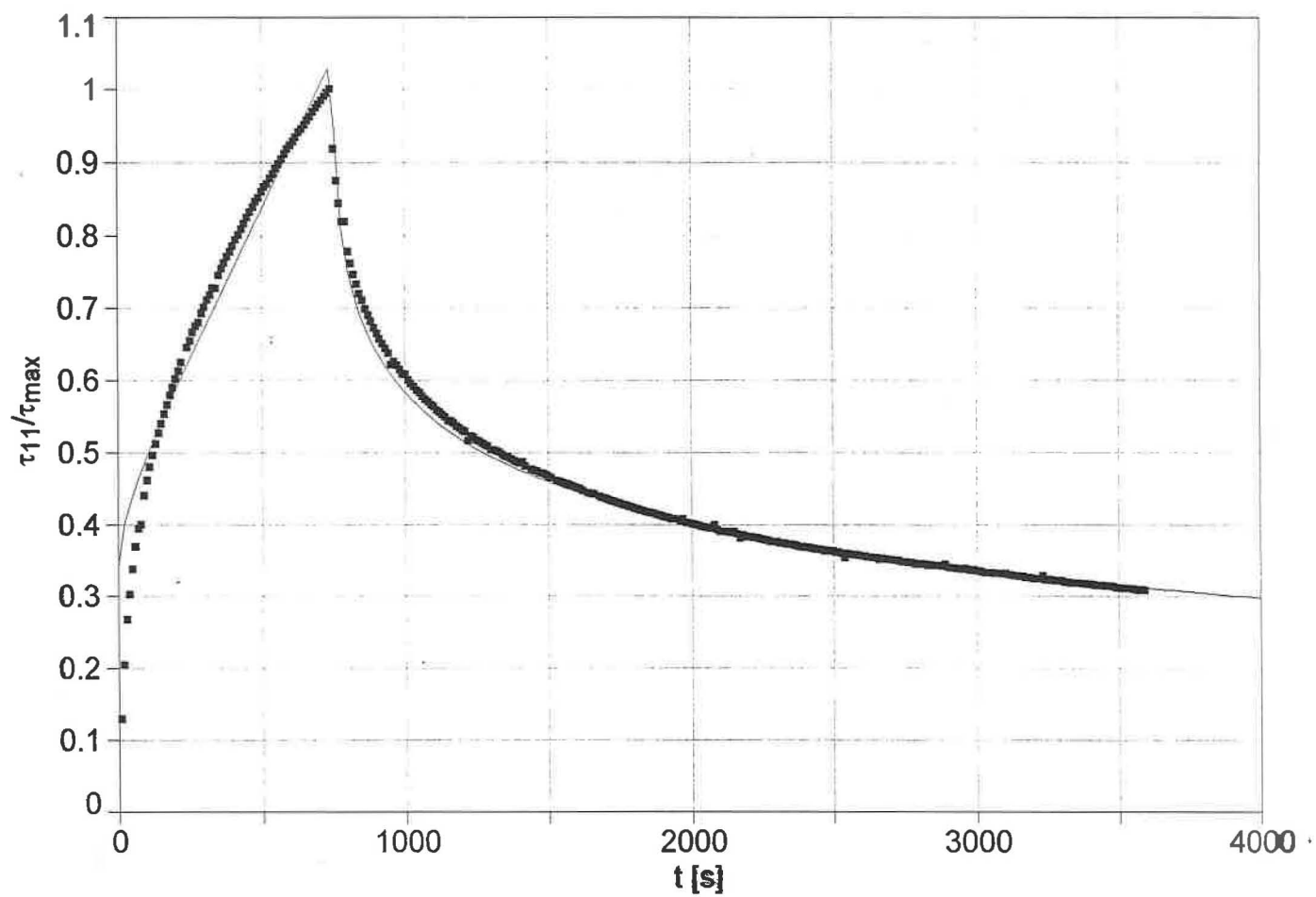


FIGURE 29. Reduced Extra-Stress Component, Sample 5(1)

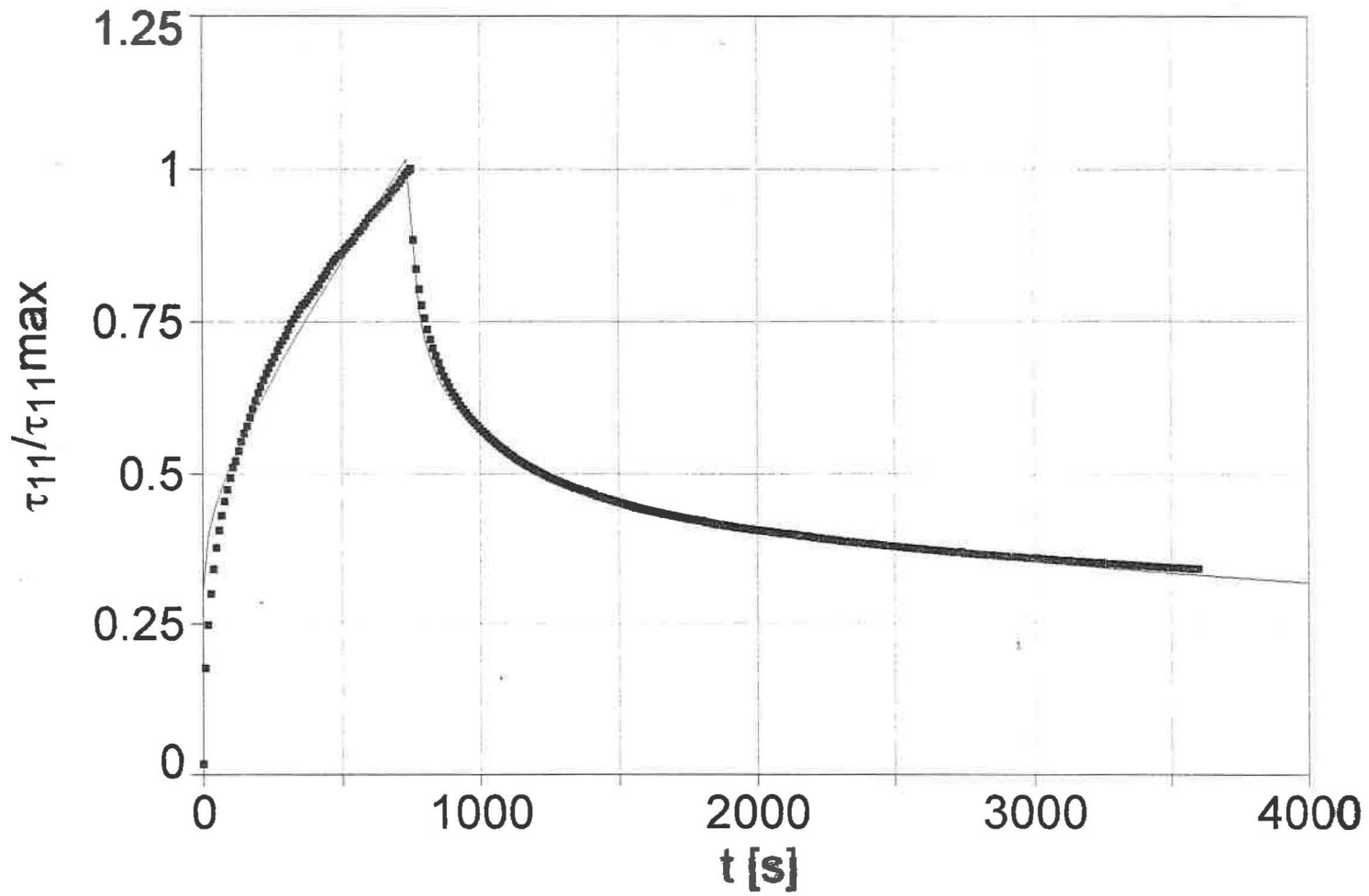


FIGURE 30. Reduced Extra-Stress Component, Sample 5(2)

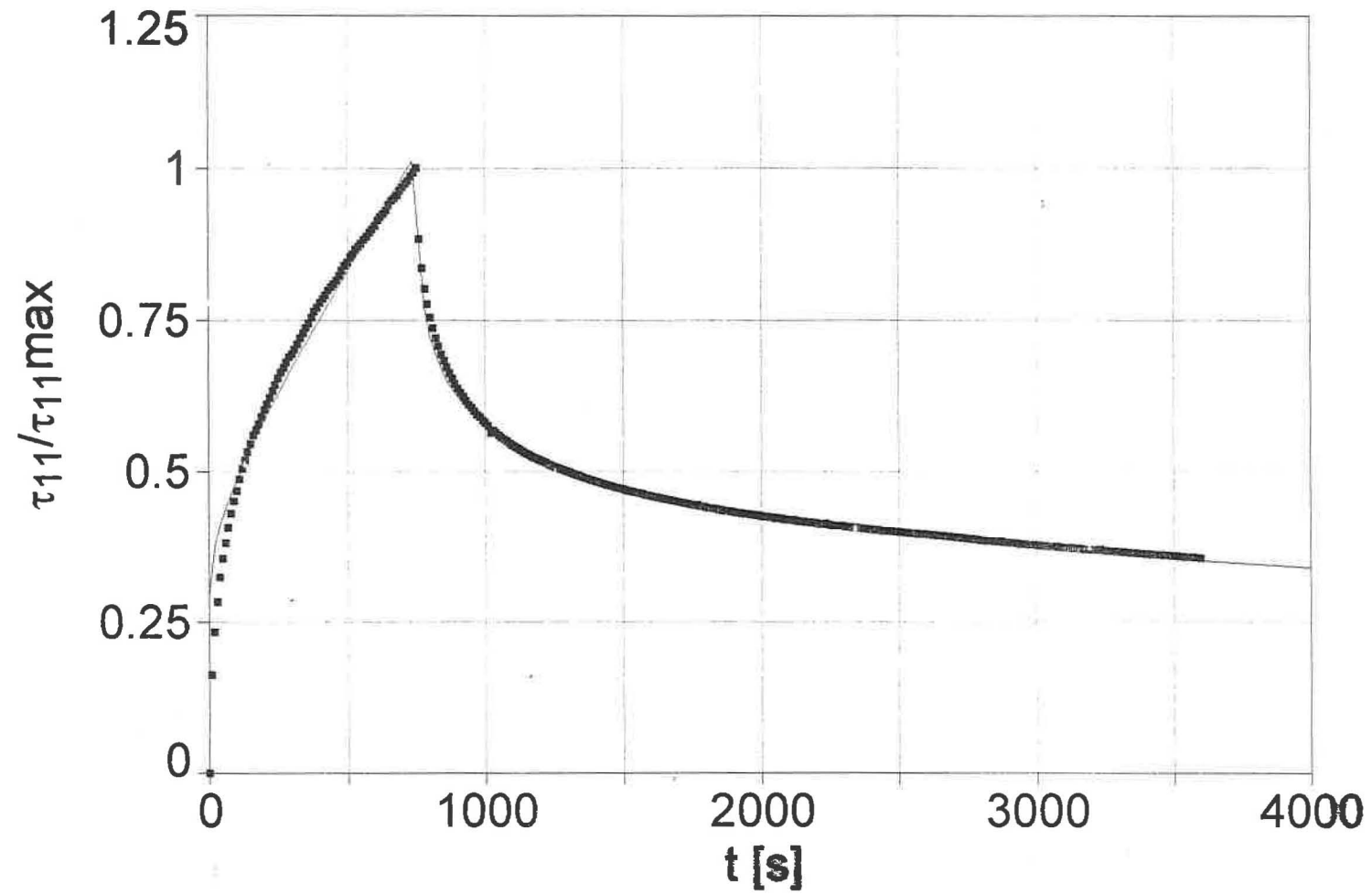


FIGURE 31. Reduced Extra-Stress Component, Sample 2379

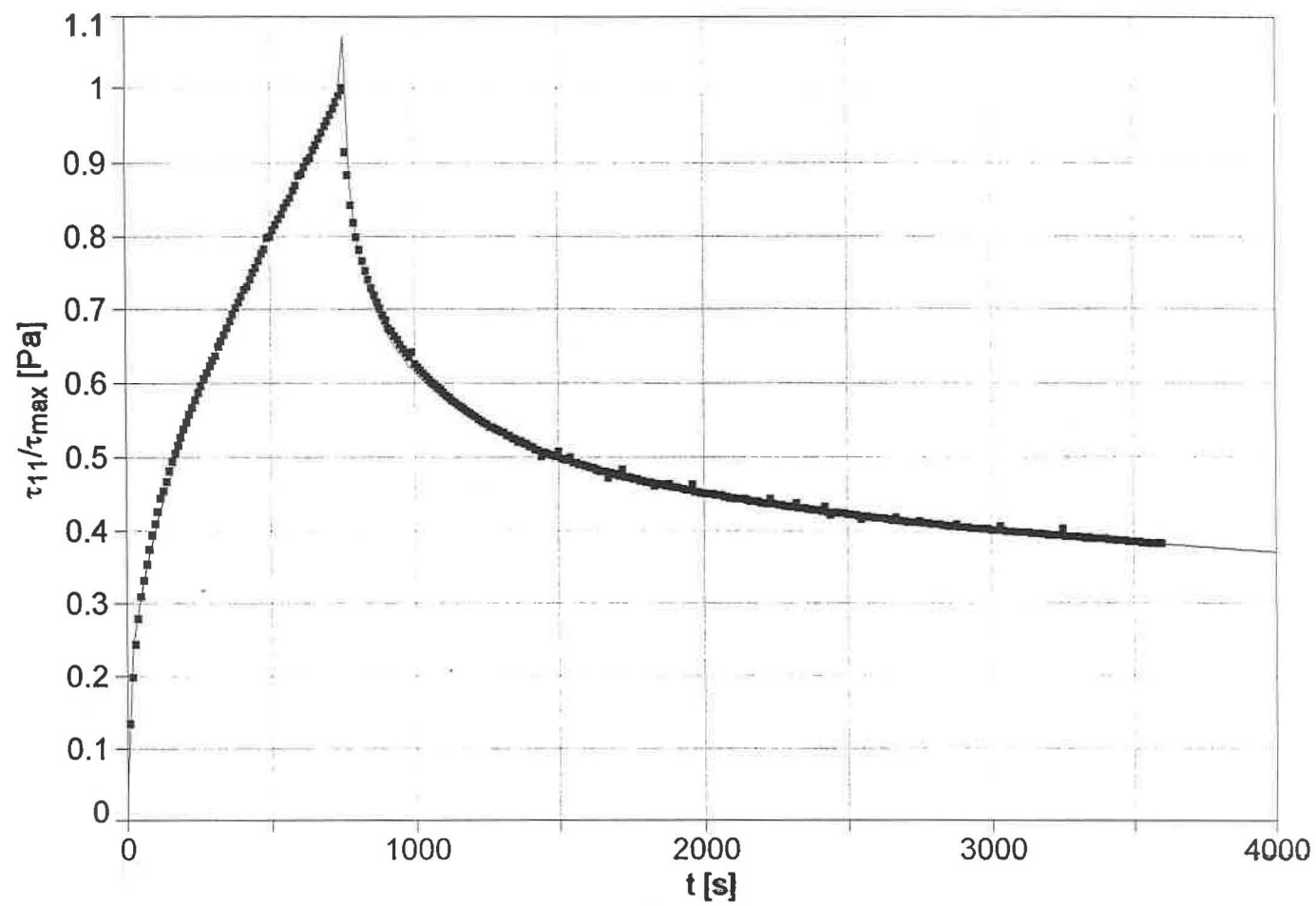


FIGURE 32. Reduced Extra-Stress Component, Sample 12(2)

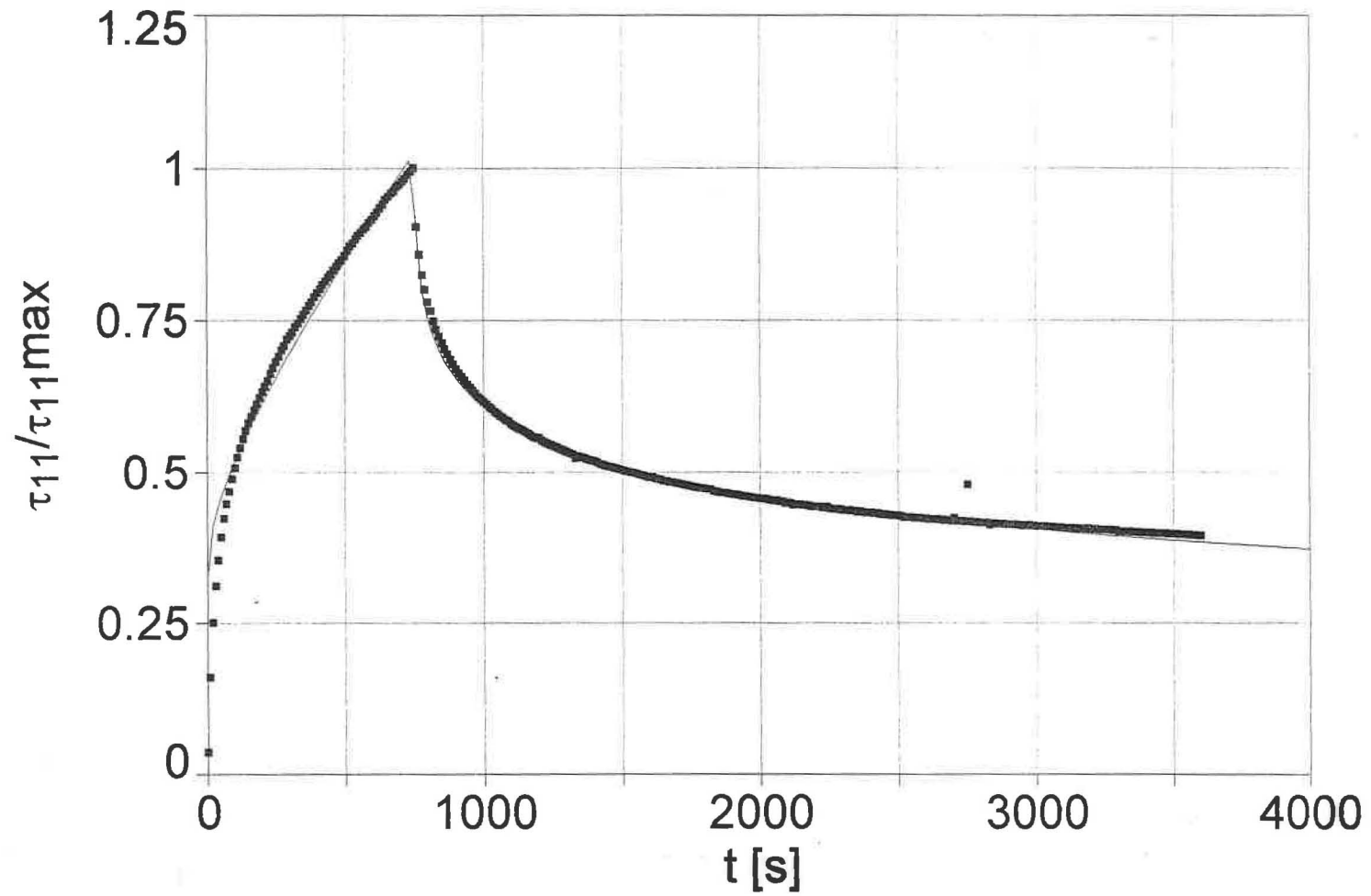


FIGURE 33. Reduced Extra-Stress Component, Sample 12(1)

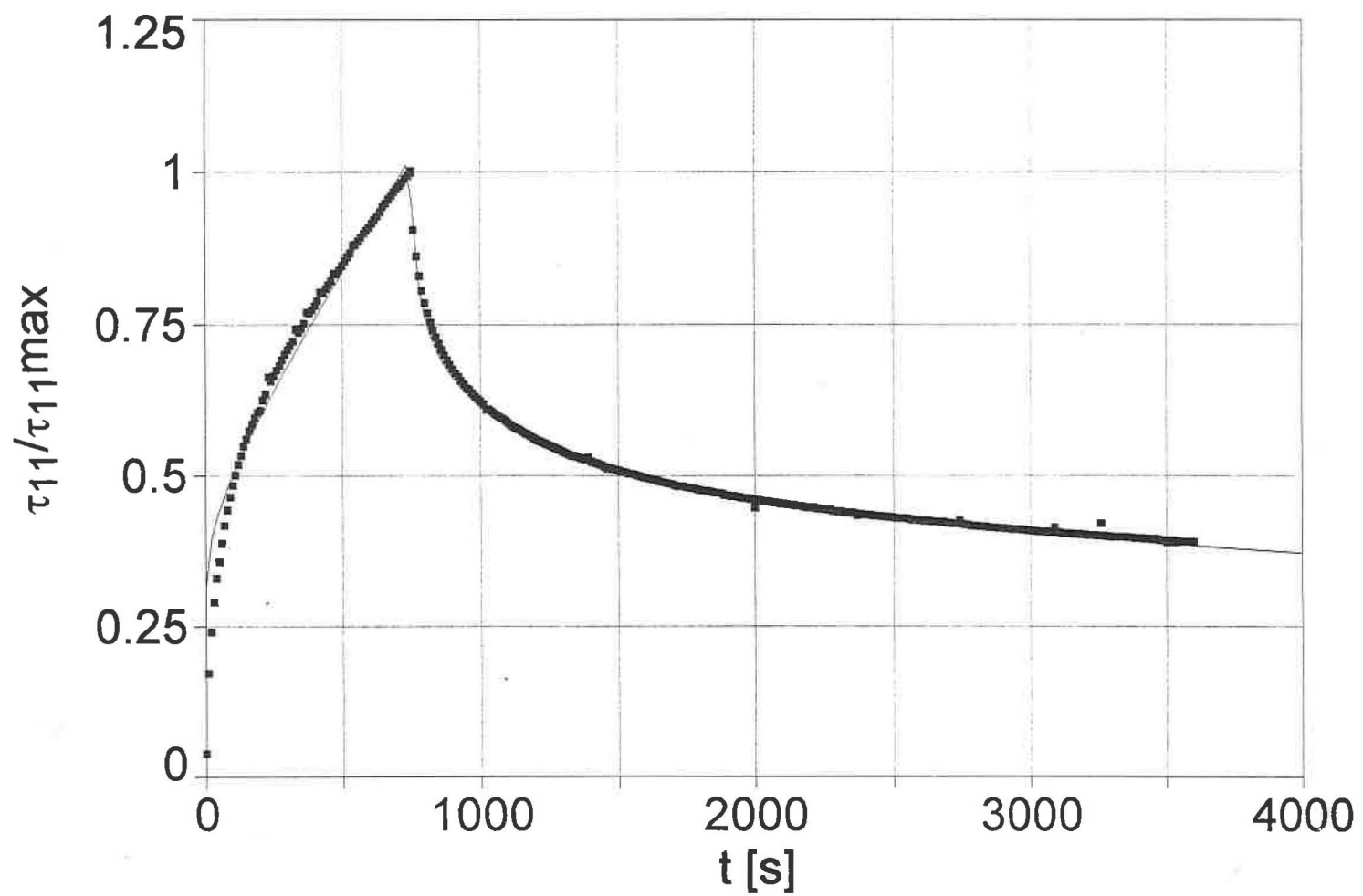


FIGURE 34. Reduced Extra-Stress Component, Sample 2450

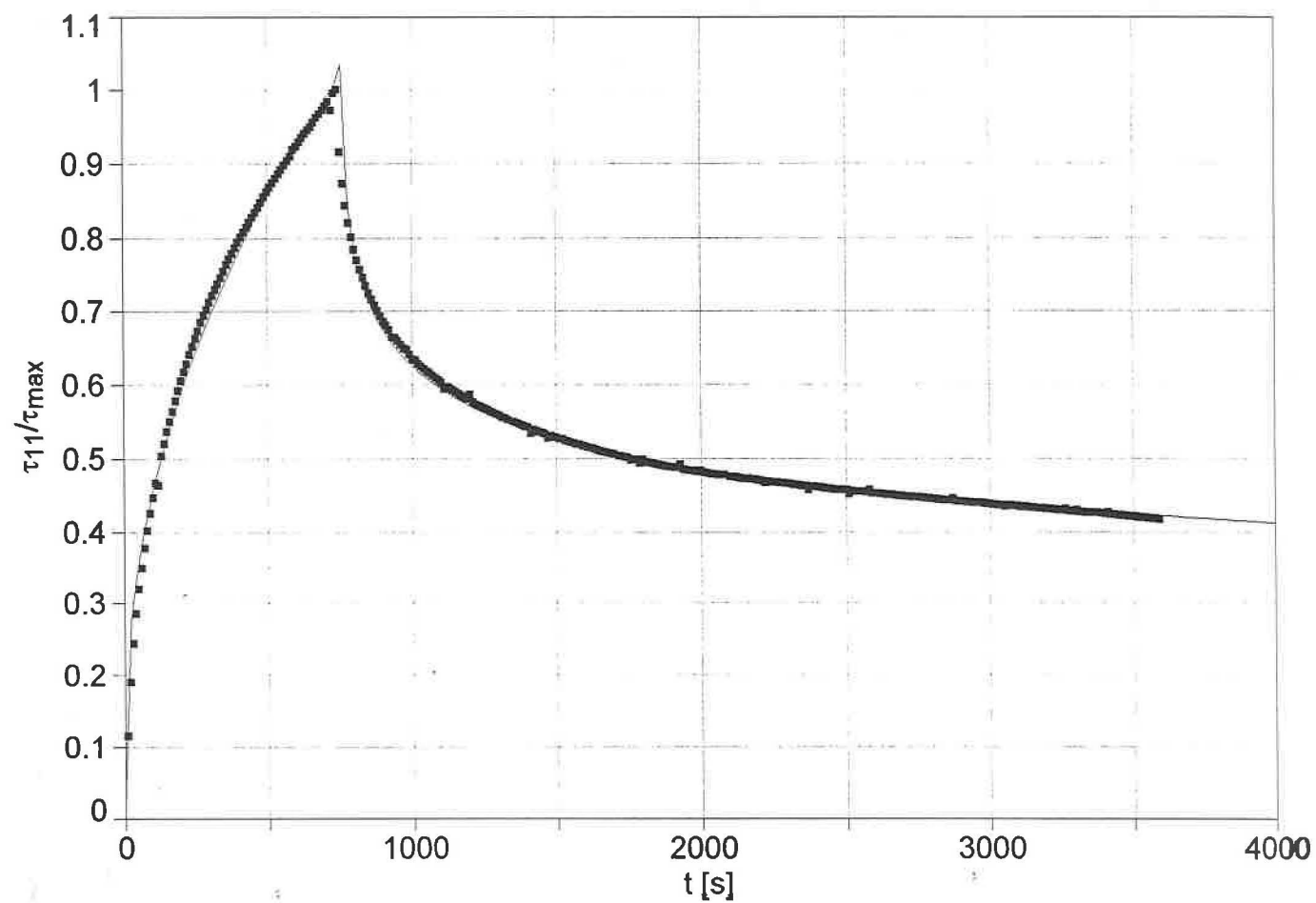


FIGURE 35. Reduced Extra-Stress Component, Sample 2380

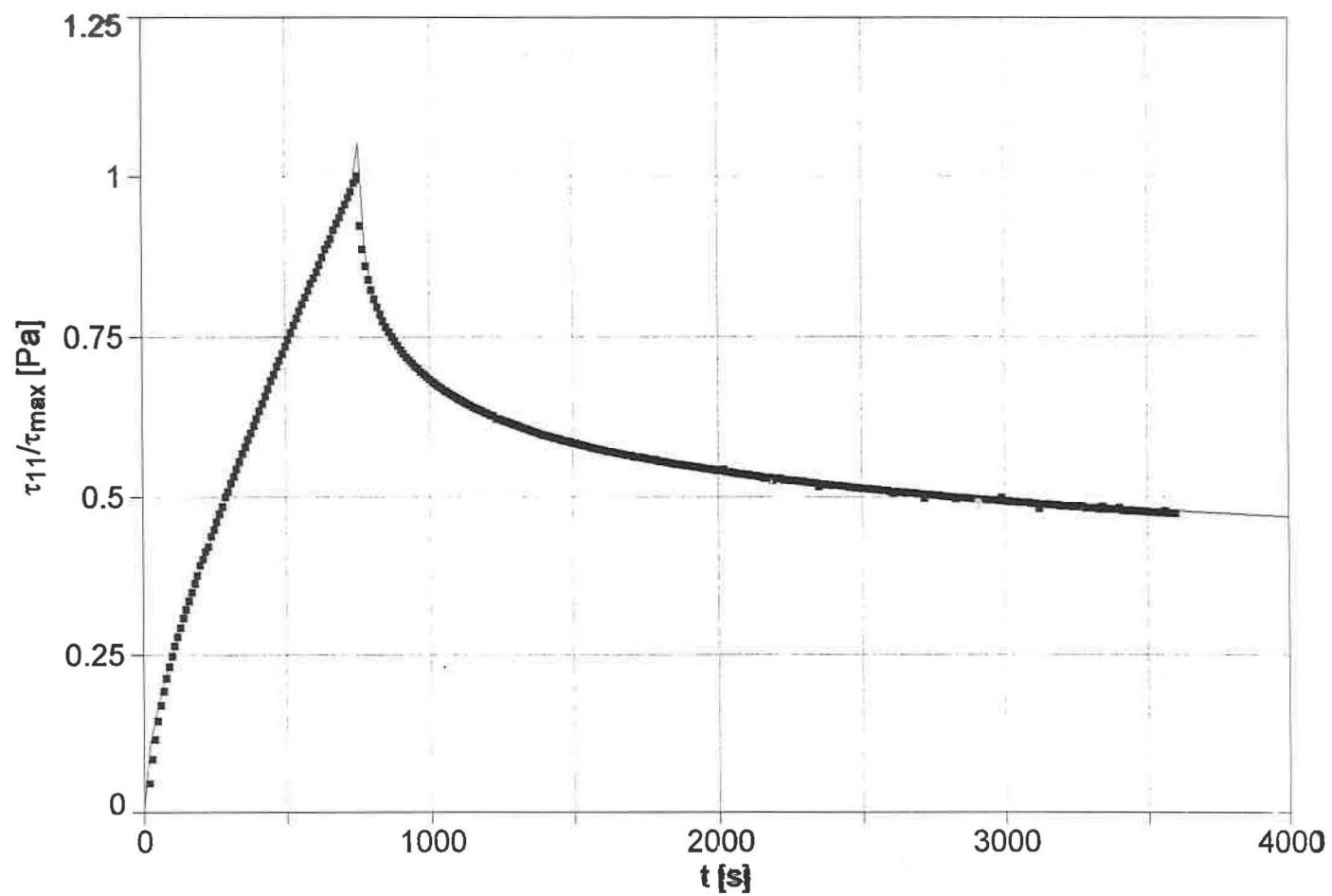


FIGURE 36. Reduced Extra-Stress Component, Broken Sample 3(1)

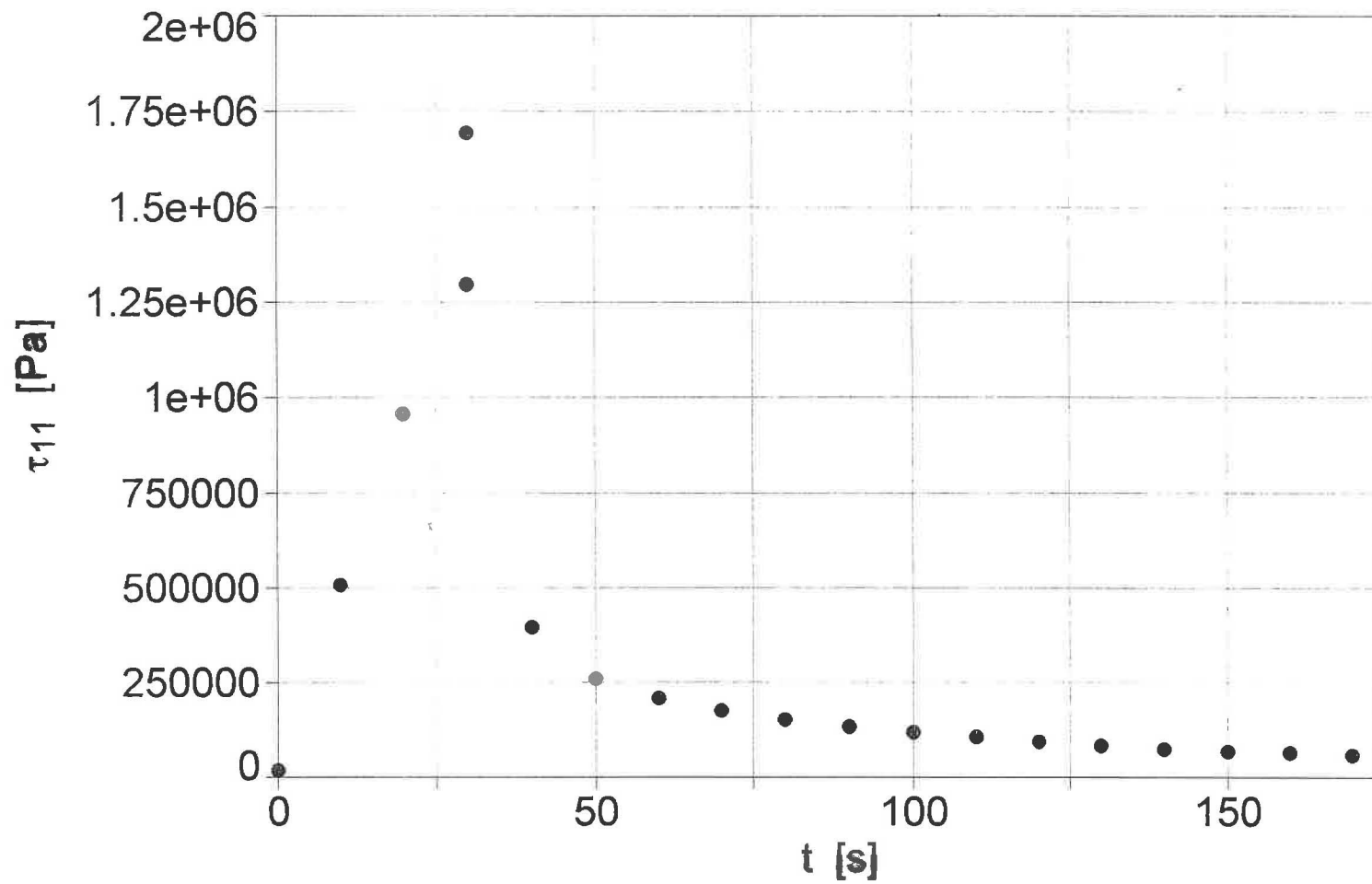


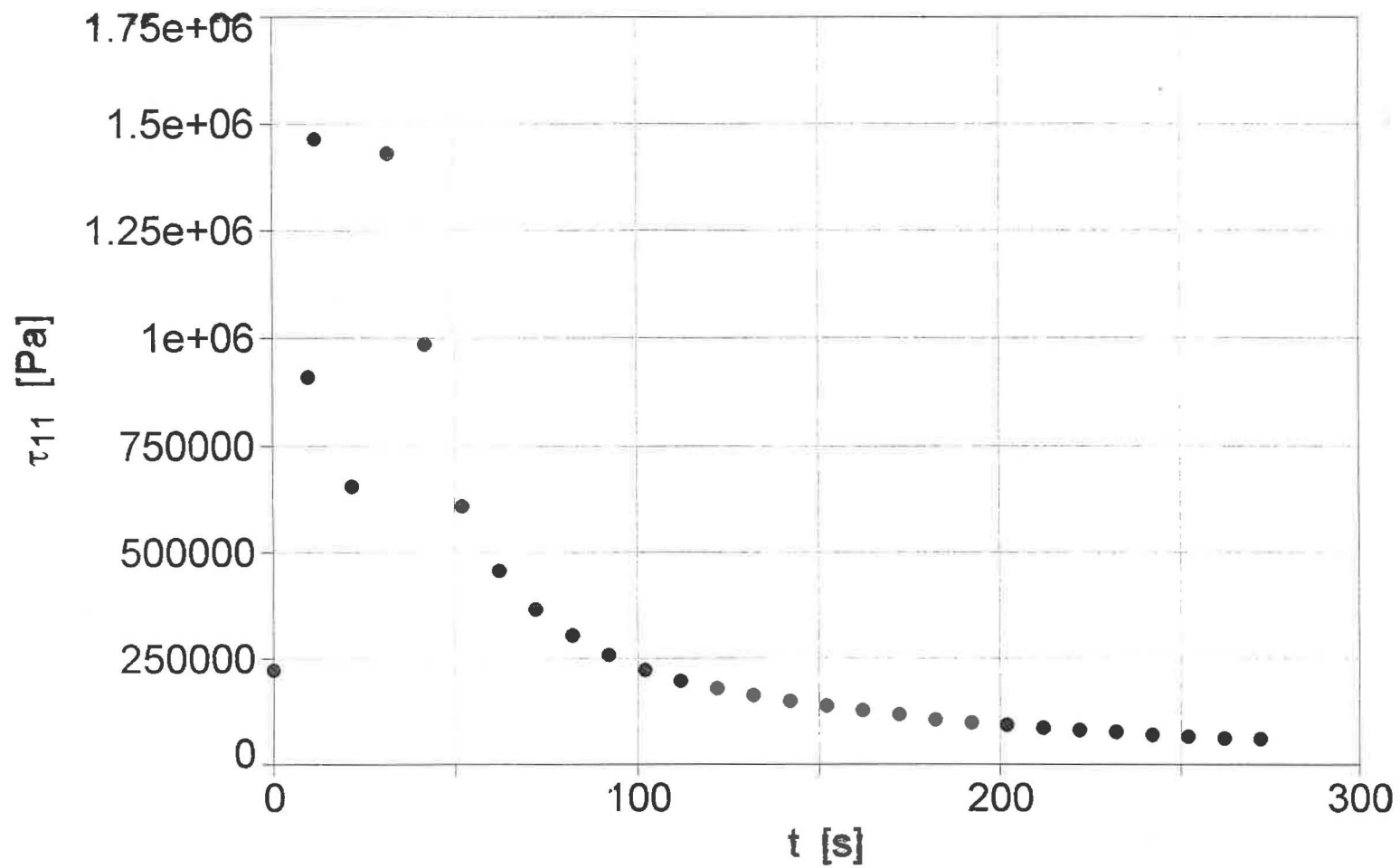
FIGURE 37. Reduced Extra-Stress Component, Broken Sample 3(2)

FIGURE 38. Reduced Extra-Stress Component, Broken Sample 9(1)

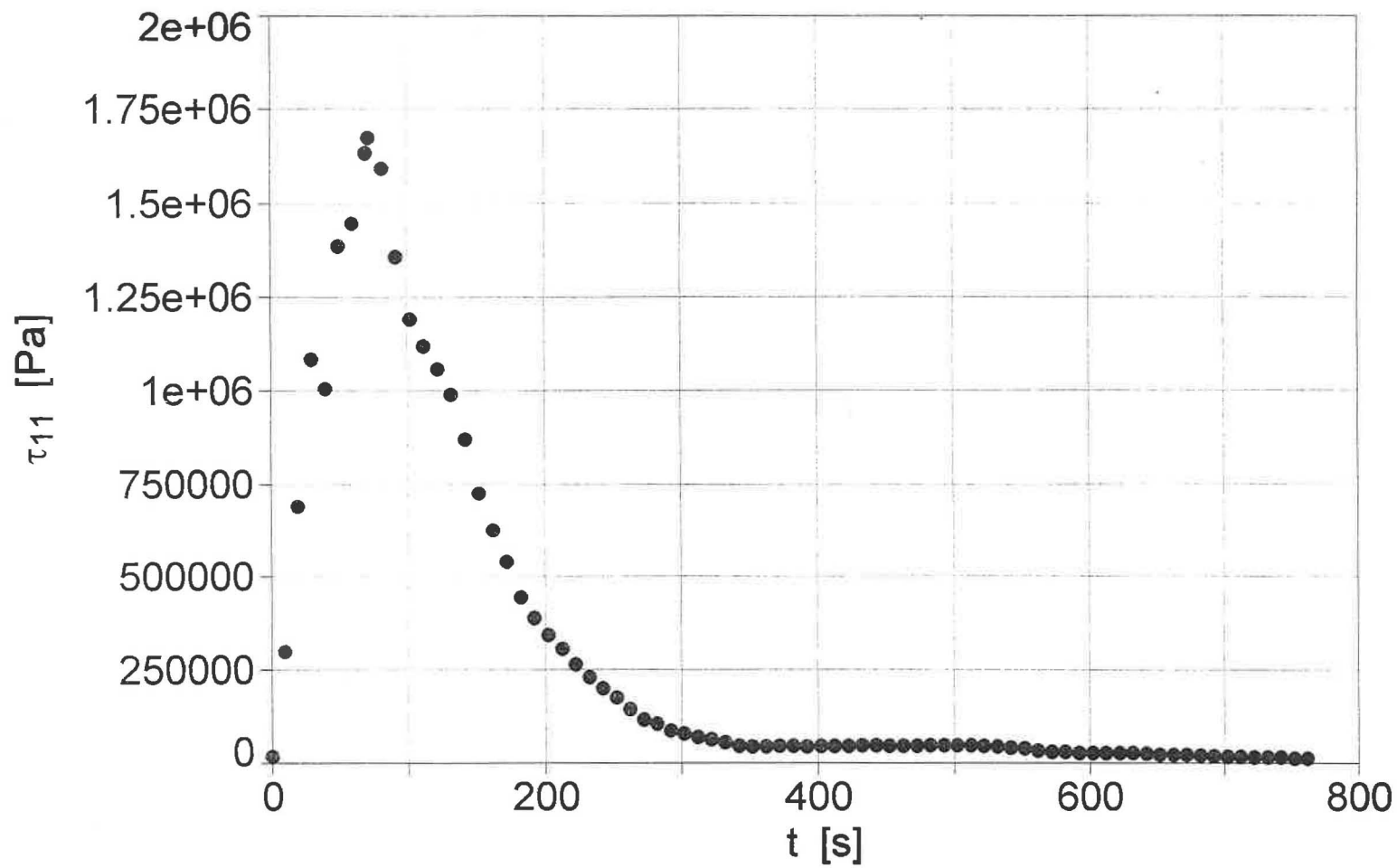


FIGURE 39. Reduced Extra-Stress Component, Broken Sample 9(2)

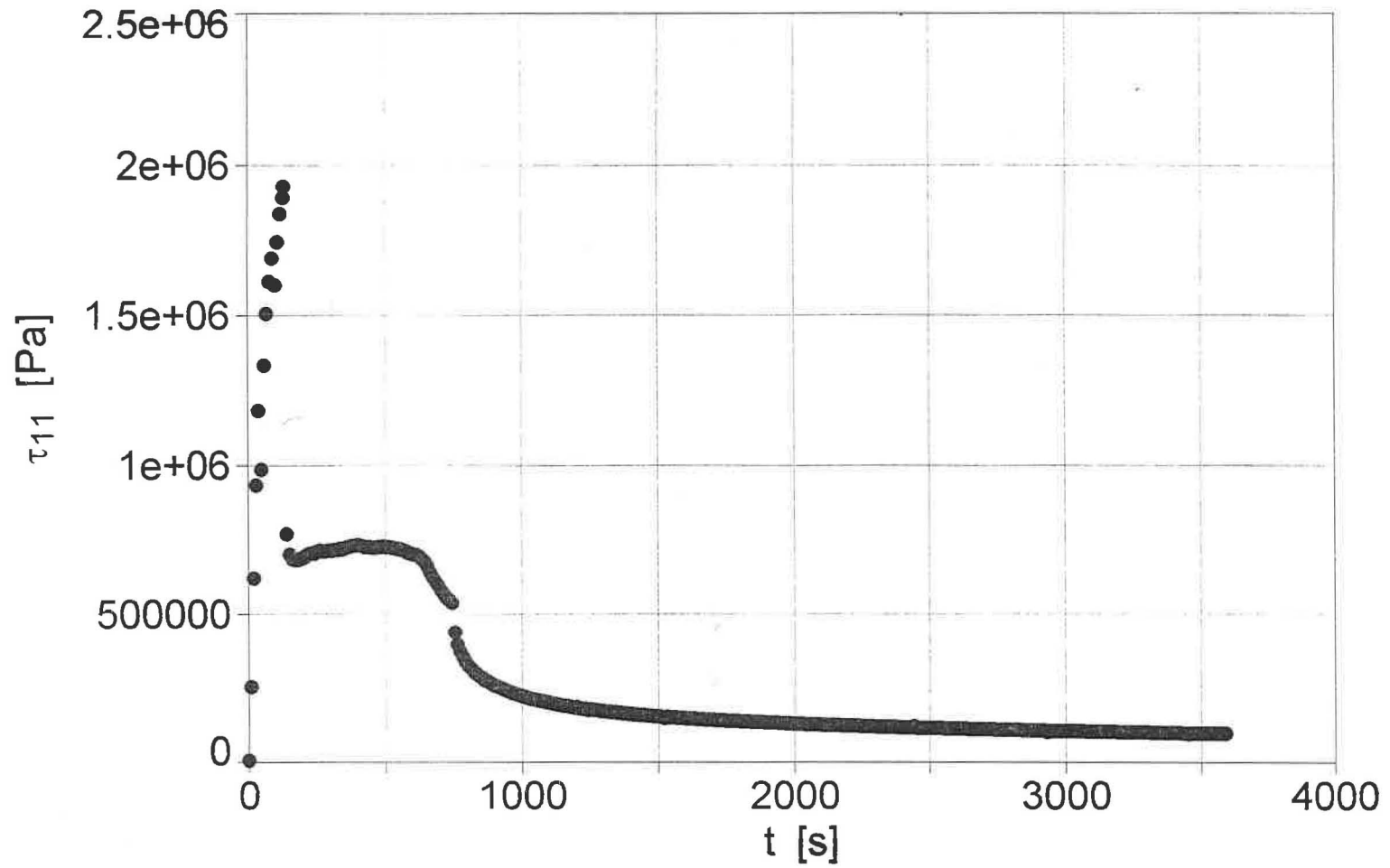


FIGURE 40. Reduced Extra-Stress Component, Broken Sample 7(2)

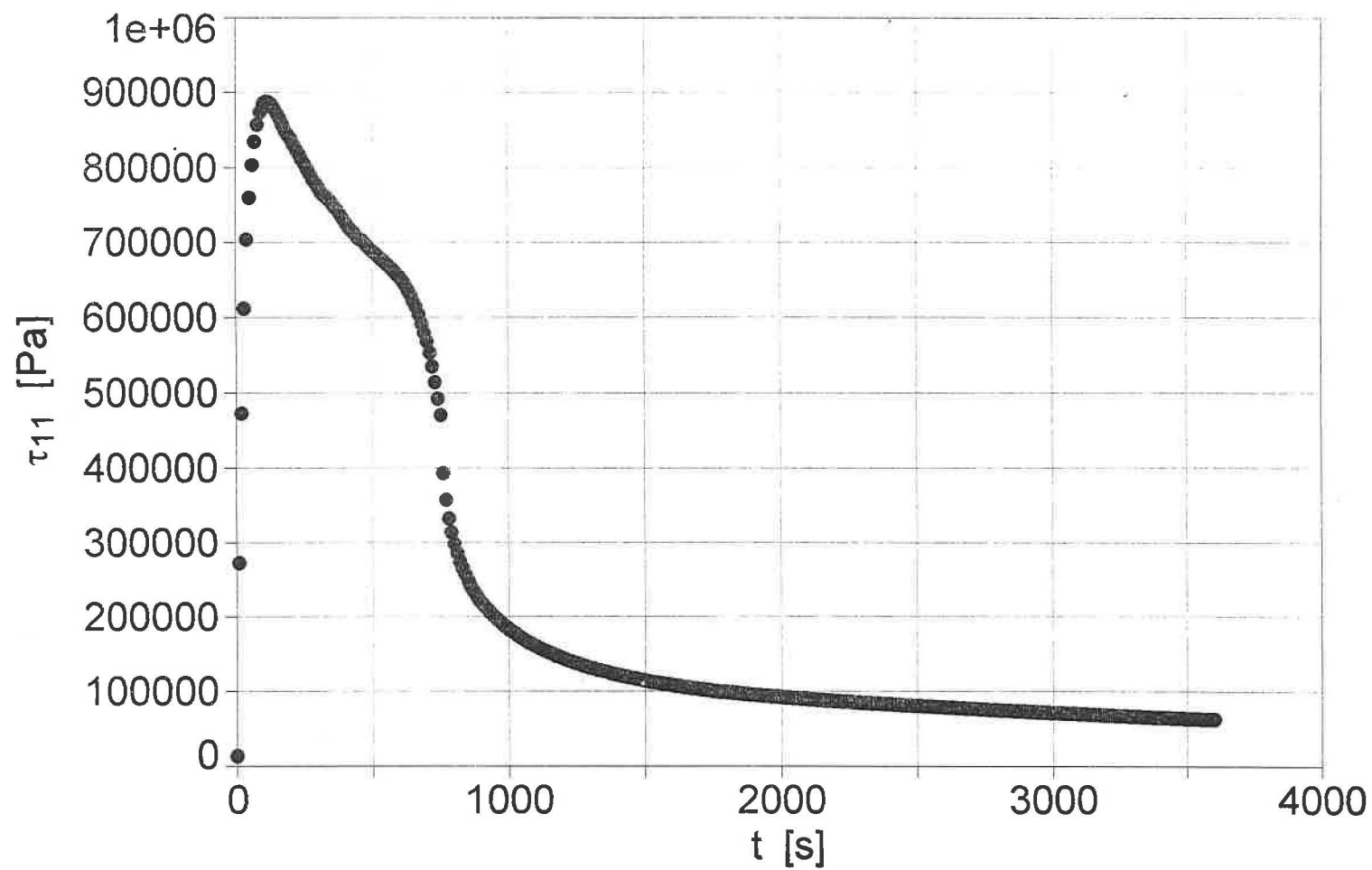


FIGURE 41. Reduced Extra-Stress Component, Broken
Sample 13(2)

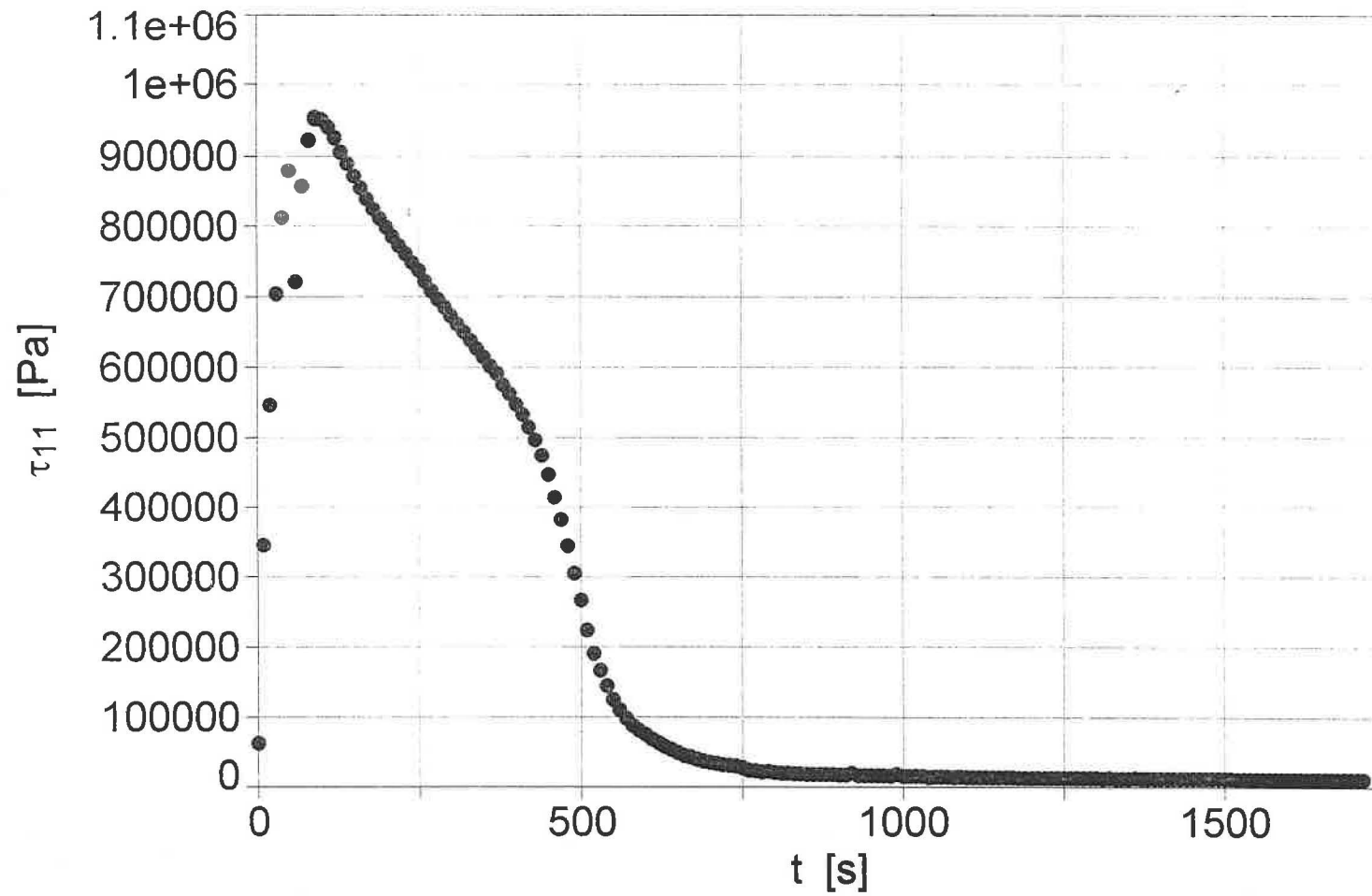


FIGURE 42. Reduced Extra-Stress Component, Broken Sample 13(3)

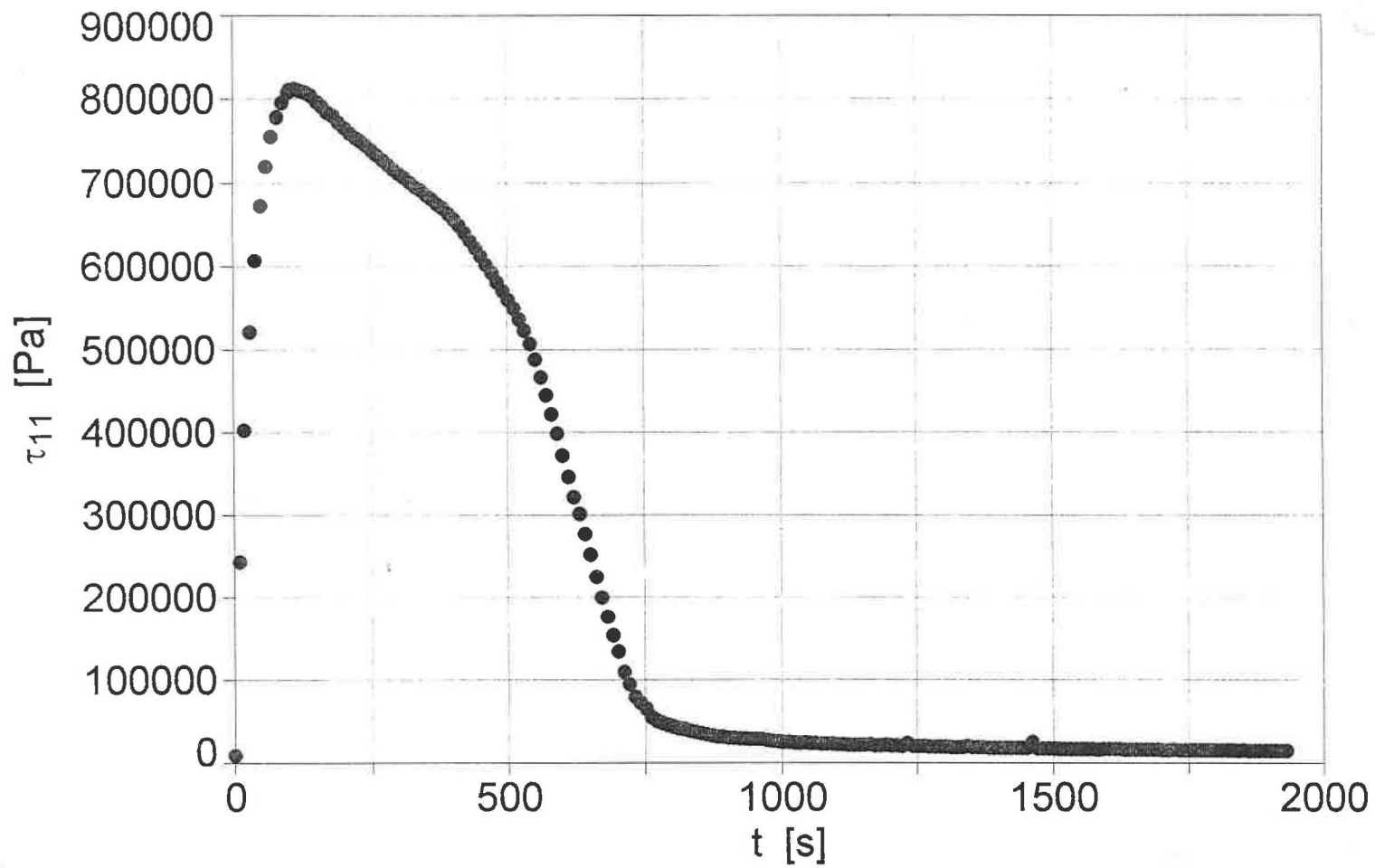


FIGURE 43. Reduced Extra-Stress Component, Broken Sample 7(2)

