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AD-A237 784

REPORT DOCUMENTATION PAGE

1a.	1b. RESTRICTIVE MARKINGS
2a. SECURITY CLASSIFICATION AUTHORITY	3. DISTRIBUTION/AVAILABILITY OF REPORT UNLIMITED
2b. DECLASSIFICATION/DOWNGRADING SCHEDULE	
4. PERFORMING ORGANIZATION REPORT NUMBER(S)	5. MONITORING ORGANIZATION REPORT NUMBER(S) AFOSR-TR- 91 0577
6a. NAME OF PERFORMING ORGANIZATION REGENTS OF THE UNIVERSITY OF CALIFORNIA 6b. OFFICE SYMBOL (If applicable)	7a. NAME OF MONITORING ORGANIZATION Same as 8a
6c. ADDRESS (City, State and ZIP Code) DEPARTMENT OF MECHANICAL, AERONAUTICAL AND MATERIALS ENGINEERING DAVIS, CA 95616-5294	7b. ADDRESS (City, State and ZIP Code) Same as 8c
8a. NAME OF FUNDING/SPONSORING ORGANIZATION AFOSR 8b. OFFICE SYMBOL (If applicable) NE	9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER AFOSR-89-0287
8c. ADDRESS (City, State and ZIP Code) BUILDING 410 BOLLING AFB DC 20332-6448 ATTN: DR. ALAN H. ROSENSTEIN	10. SOURCE OF FUNDING NOS. PROGRAM ELEMENT NO.: 61102F PROJECT NO.: 2306 TASK NO.: A1 WORK UNIT NO.:
11. TITLE (Include Security Classification) STRENGTHENING MECHANISMS, CREEP AND FATIGUE...	
12. PERSONAL AUTHOR(S) AMIYA K. MUKHERJEE AND JEFFREY C. GIBELING	
13a. TYPE OF REPORT ANNUAL 13b. TIME COVERED FROM 2/1/90 TO 1/31/91	14. DATE OF REPORT (Yr., Mo., Day) 91 MAY 21 15. PAGE COUNT 23
16. SUPPLEMENTARY NOTATION	

17. COSATI CODES			18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)
FIELD	GROUP	SUB. GR.	NIOBIUM, REFRACTORY METALS, DISPERSION HARDENING, INTERNAL OXIDATION, CREEP, LOW CYCLE FATIGUE, FATIGUE

19. ABSTRACT (Continue on reverse if necessary and identify by block number)

The creep properties of Nb-1% Zr alloy was investigated at intermediate temperatures. A model is proposed based on the operation of two parallel mechanisms. The first mechanism is based on dislocation glide-controlled creep and controls creep rate at higher stresses where the influence of the threshold stress due to the particles is negligible. At lower stresses (below 135 MPa), the threshold stress of the particles come into play and becomes the rate-controlling mechanism. The resultant effect of the operation of these two parallel mechanisms produced quite satisfactory correlation between theoretical predictions and experimental data. We have also studied the cyclic deformation of commercially pure niobium and Nb-1Zr at ambient temperature. We have conducted tests at slow strain rates, where these materials exhibit high temperature dislocation glide kinetics, and at fast strain rates, where the behavior is characteristic of the low temperature regime. We are presently exploring additional aspects of the fatigue response using a Bauschinger analysis and by testing at very low plastic strain amplitudes to see if a fatigue limit is observed.

20. DISTRIBUTION/AVAILABILITY OF ABSTRACT UNCLASSIFIED/UNLIMITED ☐ SAME AS RPT. ☐ DTIC USERS ☐	21. ABSTRACT SECURITY CLASSIFICATION Unclassified	
22a. NAME OF RESPONSIBLE INDIVIDUAL Rosenstein	22b. TELEPHONE NUMBER (Include Area Code) 202-767-4933	22c. OFFICE SYMBOL NE

DD FORM 1473, 83 APR EDITION OF 1 JAN 73 IS OBSOLETE.

SECURITY CLASSIFICATION OF THIS PAGE

ANNUAL TECHNICAL REPORT

for

Grant No. AFOSR 89-0287

for the period

February 1, 1990 to January 31, 1991

Strengthening Mechanisms, Creep and Fatigue Processes in Dispersion Hardened Niobium Alloy

submitted to:

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May 1, 1991

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CREEP AND LOW CYCLE FATIGUE BEHAVIOR OF NIOBIUM ALLOYS

RESEARCH SUMMARY

The creep properties of Nb-1% Zr alloy that was processed in a manner that produced particle-dispersed microstructure was investigated at intermediate temperatures. A model is proposed based on the operation of two parallel mechanisms. The first mechanism is based on dislocation glide-controlled creep and controls creep rate at higher stresses where the influence of the threshold stress due to the particles is negligible. At lower stresses (below 135 MPa), the threshold stress of the particles come into play and becomes the rate-controlling mechanism. The resultant effect of the operation of these two parallel mechanisms produced quite satisfactory correlation between theoretical predictions and experimental data.

We have also studied the cyclic deformation of commercially pure niobium and Nb-1Zr at ambient temperature. We have conducted tests at slow strain rates, where these materials exhibit high temperature dislocation glide kinetics, and at fast strain rates, where the behavior is characteristic of the low temperature regime. We are presently exploring additional aspects of the fatigue response using a Bauschinger analysis and by testing at very low plastic strain amplitudes to see if a fatigue limit is observed. These results represent the first basic investigation of the cyclic deformation of polycrystalline niobium.

1. INTRODUCTION

The focus of this research program is on the development and characterization of refractory metals for advanced aerospace structural applications at elevated temperatures. These applications include space nuclear power reactors and hypersonic vehicles such as the National Aerospace Plane (NASP). Compared to alternative candidate materials systems such as titanium aluminide composites, carbon-carbon composites and ceramic matrix composites, the refractory metals offer substantially better ductility and easier formability. Niobium has been chosen because it has the lowest density among the refractory metals, although its strength at elevated temperatures is well below the desired design goals. The focus of our work to improve the strength of this material is on the role of dispersed, inert ceramic particles of ZrN or ZrO_2 produced by internal nitriding or oxidizing. Our specific investigation involves studies of the kinetics of internal oxidation and nitridation of Nb-1Zr and the creep and low cycle fatigue behavior of dispersion strengthened niobium. The introduction of dispersoids provides an attractive alternative to other strengthening methods such as solid solution strengthening, which is not effective at the temperatures of interest, and fiber reinforcing, which is limited by interfacial reactions between the matrix and the fibers.

During the second year of this program, our creep studies have focussed on the behavior of Nb-1Zr alloy that contains a particle-dispersed microstructure. We have acquired a substantial amount of creep data for this material at intermediate temperatures. The results are described by a model based on dislocation glide controlled creep at high stresses operating in parallel with a mechanism dominated by a threshold stress at low stresses. We have completed our low cycle fatigue studies of the baseline alloys (commercially pure Nb and Nb-1Zr), and designed the specimens for testing dispersion strengthened material. In the following sections, we describe our progress during the past year in greater detail and outline our research plans for the third year of our program.

2. RESEARCH HIGHLIGHTS

During the second year of our program, we have:

(1) Developed the equipment, software and procedures to conduct monotonic and high resolution stress change creep tests at temperature up to 1300K in vacuum.

(2) Measured the monotonic creep properties of Nb-1Zr processed in a manner to produce a particle-dispersed microstructure. Stress change tests were used to verify that the material is particle strengthened and that softening occurs during deformation.

(3) Developed a model of creep deformation that describes the results for particle strengthened Nb-1Zr. This model is based on parallel mechanisms of dislocation glide controlled behavior at high stresses and threshold stress dominated behavior at low stresses.

(3) Completed the baseline low cycle fatigue studies of commercially pure polycrystalline Nb and Nb-1Zr. We have used low strain rates to determine high temperature behavior and high strain rate to assess low temperature characteristics. Cyclic hardening is observed and there is a microplastic plateau in the pure Nb. In addition, the effect of strain rate suggests that deformation of the pure Nb is controlled by the Peierls stress acting on gliding screw dislocations.

(4) Shown that Nb-1Zr is stronger in cyclic deformation than Nb, with little influence of strain rate. Deformation at both strain rates is controlled by the interaction between gliding edge dislocations and solute atoms.

3. MATERIALS AND EXPERIMENTAL PROCEDURES

3.1 Materials

The niobium alloys that are being used in this study were supplied by Teledyne Wah Chang. The alloys were processed by compacting powder, arc melting, machining, forging, and rolling sheet or extruding rod. The average total impurity content of these materials is approximately 2300 ppm. The Nb-1Zr sheet used in the creep studies was annealed for two hours at 1477K in vacuum (6.6×10^{-6} Pa), which produced an average grain size of approximately 14.3 μm. The niobium and niobium alloy rods for low cycle fatigue were annealed at 1473K for 1 hour at Teledyne Wah Chang to relieve the strain from working.

Tensile creep specimens were punched from 1 mm sheet using a custom made punch and die set, and tested without further heat treatment. Cylindrical specimens for low cycle fatigue testing were machined from 1.6 cm diameter bars according to ASTM standard E606-80. Both commercially pure niobium (CPNb) and Nb-1Zr of compositions similar to those for the sheet material were tested in the as-received condition. Prior to testing, each specimen was mechanically polished to a smooth surface finish as provided by 1 micron diamond paste.

3.2 Creep Testing

The creep test were performed in an apparatus purchased from Applied Test Systems (ATS) with funding obtained from a previous AFOSR equipment grant. To heat the samples to the test temperature, a single zone resistance furnace was used. All tests were done in vacuum at pressures ranging from 2×10^{-3} to 6.6×10^{-4} Pa. The deformation of the samples was measured with a super linear variable capacitor (SLVC) which has a resolution of 2.5×10^{-5} mm. The force acting on the specimen was measured with a load cell which was mounted directly in the load train. The two signals, force and displacement, were sent to an A/D convertor and from this to a personal computer. With this system, it is possible to measure (true) strains up to 54% with a resolution of approximately 1×10^{-5}.

3.3 Low Cycle Fatigue Testing

Constant plastic strain amplitude low cycle fatigue tests were performed using procedures described in our previous progress report. Testing was conducted at two different constant plastic strain rates, defined as $\dot{\epsilon}_{pl} = 2\nu\epsilon_{pl}$ = constant [3]. The low strain rate was $2 \times 10^{-4}\ s^{-1}$; the high strain rate was $2 \times 10^{-2}\ s^{-1}$. All tests were carried out using a computer controlled MTS servohydraulic testing system purchased under a previous AFOSR sponsored equipment grant.

4. RESULTS OBTAINED DURING THE SECOND YEAR

4.1 Equipment Upgrades

Several important modifications were made to the creep testing apparatus in order to enable us to conduct our experiments. Although this was a commercially produced system, it was necessary to improve the vacuum seals, add additional external cooling, incorporate internal cooling for the SLVC and modify the grip and specimen design in order for the equipment to operate properly at the test temperatures of interest. A 5000 lb. capacity load cell was also added to enable us to accurately monitor the load during monotonic and stress change tests. The latter tests are particularly important to our efforts to understand the mechanisms of dislocation/dispersoid interactions. For these tests to be successful, it is important that the transient response of the material be recorded with high strain, load and time resolution. For this reason, we have also added a new high speed 16 bit digital-to-analog (A/D) convertor to our data acquisition system and developed the software necessary to acquire and analyze the creep data.

4.2 Monotonic Creep Testing

A set of creep curves for the Nb-1Zr alloy tested at 1300K and various stresses is shown in Figure 1. In this figure, the data are plotted as creep rate, $\dot{\epsilon}$, as a function of (true) strain, ϵ. It is more suitable to plot the results in this manner than in the usual strain-time plots, because the steady state or minimum creep rates show up more clearly. One can see that for all stresses the primary creep transients show normal (pure metal type) behavior.

The length of the primary creep transients is dependent on stress: the smaller the stress the shorter the transients become. Further, the creep behavior can be separated into two distinct regimes:

a) Stresses above 150 MPa:
At stresses above 150 MPa the primary creep transients are followed by more or less pronounced regions of steady state creep. These regions are followed by the so-called region III where necking and finally failure of the samples occurs.

b) Stresses below 150 MPa:
At these stresses a minimum in the creep curves is observed. These minima become more pronounced with decreasing stress. In addition, the minima shift towards smaller strains. After the minimum, the creep rate increases slightly until region III is reached and necking occurs.

In Figure 2 the minimum creep rates are plotted as a function of stress in the conventional double-logarithmic manner. The horizontal bars indicate errors in the stress determination caused by uncertainties in measuring the cross sectional areas of the samples. Also plotted in Figure 2 are lines (solid, dashed and dotted) which are based on creep models and which are discussed in the next section.

4.3 Modelling of the Creep Rate

The results obtained so far indicate that the investigated material is a particle strengthened alloy. It is assumed that coherent ZrO_2 dispersoids were formed during the final heat treatment at Teledyne Wah Chang. The assumption that the material is particle strengthened is supported by a stress change test where the stress was increased after about 20% strain (see Figure 3). A comparison of the stress change result with two monotonic tests at the same stresses clearly shows that the creep rates from the cycling test lie above the monotonic curves, indicating that softening had occurred at the low stresses.

We propose that a simple model can be used to describe the creep results. The model consists of two parts which are connected in such a manner that the faster one dominates. The first mechanism is glide controlled creep which should dominate at stresses above 135 MPa where the influence of the threshold stress due to the particles is negligible. This mechanism is represented by the following equation:

$$\dot{\epsilon}_1 = A_{ex}\,\sigma^{n_{eff}} \sinh\left(\frac{V\sigma}{MkT}\right) \exp\left(\frac{-Q}{RT}\right)$$

At stresses below 135 MPa the threshold stress of the particles comes into play and the second mechanism is now the dominant process:

$$\dot{\epsilon}_2 = A_{tc}\left(\sigma - \sigma_p\right)^{n_o} \exp\left(\frac{-Q}{RT}\right)$$

The parameters in the above equations have the following meanings:

σ = Applied stress
A_{tc} = Scaling factor
σ_p = Threshold stress
n_o = Stress exponent of the matrix (pure Nb)
Q = Activation energy
R = Gas constant
A_{ex} = Scaling factor
n_{eff} = Effective stress exponent
V = Activation volume
M = Taylor factor
k = Boltzmann's constant
T = Temperature

For the threshold stress a value of 20 MPa was taken, derived from a Lagneborg-Bergman plot where the creep rate to the power of the inverse matrix stress exponent (of pure Nb, n_o=3) is plotted as a function of stress. For the activation volume a value of 450 b^3 was used (this value was estimated from a plot of the stress exponent n as a function of $\sigma/(MkT)$ for our specimen at 1300K). As one can see in Figure 2, the model describes the measured minimum creep rates in a fairly good manner.

4.4 Internal Oxidizing

An important aspect of our original proposal was to internally oxidize and internal nitride the Nb-1Zr alloy in order to produce a dispersion strengthened material. However, as noted above, the as-received sheet appears to already contain dispersoids. This conclusion is supported by the results of our attempts to internally oxidize the alloy. Specimens were annealed at 1000, 1100, 1200 and 1300K for 100 hours in a vacuum of 1.4 x 10^{-4} Pa. Based on previous work, the partial pressure of oxygen under these conditions should be sufficient to create ZrO_2 particles. Specimens of material prepared in this way were tested at 1300K. The results of these tests, illustrated in Figure 4, demonstrate that the as-received material is the strongest (lowest creep rate) and that annealing serves only to soften the material, presumably through particle coarsening.

The role of oxygen can be seen in a different way by comparing the creep result of a specimen wrapped in Nb foil with the result for an unwrapped specimen (Figure 5). The unwrapped specimen demonstrates a higher creep rate in vacuum, suggesting that oxygen aids the particle coarsening process. This results also demonstrates the improvement in creep properties that can be obtained through the use of protective coatings. It should be noted that the creep results described in the previous section were obtained from samples that were wrapped in niobium foil.

4.5 Low Cycle Fatigue Testing

The emphasis of our work to date has been on developing the baseline cyclic deformation data for commercially pure niobium (CPNb) and the Nb-1Zr alloy at room temperature. As noted in our original proposal, virtually no information is available in the literature regarding these materials in polycrystalline form. The baseline studies are now complete, and resulted in a Master of Science thesis that was submitted in September, 1990.

Our interpretation of the cyclic deformation of CPNb and Nb-1Zr is based on the mechanisms of dislocation dynamics, as described in a previous report. The data are presented in the form of cyclic stress-strain curves in Figure 6 for the two materials each tested at two strain rates. In the commercially pure niobium (CPNb), the high strain rate behavior is characteristic of low temperature deformation, while the low strain rate represents the high temperature regime. The differences in stress for the two rates arises because lattice friction (Peierls stress) limits the mobility of screw dislocations. There is a much smaller difference in stress for the Nb-1Zr tested at two rates due to the fact that the solute atoms limit edge dislocation mobility and push the low temperature friction stress controlled regime to higher strain rates (lower temperatures).

We are now extending our original analysis of the experimental data in two important ways. The first is a Bauschinger analysis aimed at separating the stress required to deform the material to a given plastic strain amplitude into a frictional component and a back stress. The friction stress characterizes the effects of lattice friction and solute atoms on dislocation glide, whereas the back stress represents the contributions of work hardening. It is our goal to relate these results to the dislocation microstructures that develop. The other way in which we are extending our earlier work is to conduct tests of CPNb at very low amplitudes at a slow strain rate. As shown in Figure 7, the cyclic stress-strain curve for the CPNb exhibits a constant stress plateau at low strain amplitudes at the high strain rate. This plateau suggests that there should be a fatigue limit, as expected for BCC metals. In contrast, the data at slow strain rates do not indicate a plateau. However, we need to complete the data by conducting low amplitude tests to confirm this possibility. The lack of a plateau at low rates, hence lack of fatigue limit, would have important engineering implications because our practical understanding of fatigue in BCC metals (including steel) is based on a fatigue limit. However, most fatigue tests are conducted at high rates to minimize testing time, whereas service conditions may impose slower loading rates where the intrinsic material behavior is different.

We have not observed any mechanical evidence that the Zr in the alloy is oxidized in the as-received material. Rather, our results for this material show a strong yield point at room temperature indicative of the solid solution hardening by Zr as expected. Of course, the creep specimens were machined from thin sheet that had been rolled by the producer, whereas the low cycle fatigue specimens were machined from extruded bar stock that had not been processed as heavily. It is important at this point to fully characterize the microstructure of the Nb-1Zr alloy using TEM in order to confirm that our material does not contain oxide particles.

5. RESEARCH PLANS FOR THE THIRD YEAR

5.1. Equipment Upgrades

We have recently purchased a set of hydraulic grips for our servohydraulic testing machine under related NSF program support. When these grips are installed, they will significantly enhance our ability to conduct the necessary low cycle fatigue experiments of dispersion strengthened Nb-1Zr.

5.2 Constant Strain Rate Tensile Tests

We plan to complete our tests of Nb, Nb-1Zr, nitrided Nb-1Zr, and oxidized Nb-1Zr at constant strain rates varying from 10^{-6} to 10^{-1} s^{-1} at elevated temperatures. The high temperature tests ($T>=1073K$) will be carried out at Lawrence Livermore National Laboratory, where suitable testing facilities are available. These experiments will be used primarily to identify the Orowan stress for deformation.

5.3 Creep Testing

We will continue our program to conduct creep tests on the Nb-1Zr alloy. In particular, we will complete monotonic tests on a new heat of material at 1300K. This material appears to be somewhat stronger than the previous heat. We will conduct monotonic tests at 1200K and temperature change tests in order to identify the activation energy for creep in this material. The temperature change tests are particularly appropriate because of microstructural instability in this material. We will also conduct a series of stress reduction tests in order to identify the mechanisms of high temperature deformation, with emphasis on identifying the threshold stress. These experimental efforts will be coupled with further developments of the appropriate models to describe the creep behavior in this material.

5.4 Low Cycle Fatigue Testing

Our work during the third year will focus on the development of techniques and procedures for preparing and testing internally oxidized Nb-1Zr at room temperature. Because the oxidizing treatment requires diffusion through the specimen thickness, it is not feasible to use the relatively large, (6.4 mm diameter) solid conventional low cycle fatigue specimens. As an alternative, our design is based on a thin-walled hollow cylindrical specimen. The relatively thin walls will enable us to diffuse oxygen into the material to create the ceramic dispersoids. We are presently working on finding an economical combination of material source and machining method to form tube specimens of this type. During the third year of our program, we plan to demonstrate the feasibility of this approach.

5.5 Transmission Electron Microscopy

In order to fully describe the creep behavior of the Nb-1Zr alloy, it is necessary to characterize the particle size, volume fraction and spacing. Further, we need to have an adequate understanding of the way in which these parameters change during deformation. For this reason, detailed transmission electron microscopy studies will be undertaken on samples of deformed material. In addition, it is necessary to investigate whether the low cycle fatigue specimens also contain particles similar to those present in the sheet material used in the creep studies. Our microscopy work will also provide us with a better understanding of the dislocation structures that develop during cycling.

Recent work by several investigators has shown that the nature of the particle-dislocation interaction strongly influences the mechanical response of the material. In particular, strong departure-side pinning forces are developed in systems in which the dispersoids are incoherent with the matrix. These attractive interactions then control the release of dislocations from particles, and are thus the rate controlling mechanism. In order to fully understand the mechanical properties of the dispersion strengthened niobium, it will be necessary to study this particle-matrix interface in detail using high resolution transmission electron microscopy (HRTEM). Although preliminary work can be done at UC Davis, the detailed study will require the use of the facilities and collaboration with researchers at the National Center for Electron Microscopy at Berkeley. During the third year of our program, we will initiate our HRTEM study of undeformed and deformed niobium containing dispersoids.

6. PERSONNEL

The following personnel are primarily in the work of this program:

Professor Amiya K. Mukherjee, Principal Investigator

Professor Jeffery C. Gibeling, Principal Investigator

Dr. Maximilian A. Biberger, Visiting Assistant Researcher

Dr. Hong Sheng Yang, Postgraduate Researcher

Mr. Michael J. Davidson, Graduate Research Assistant

Mr. John M. Meininger, Graduate Research Assistant

Mr. Jorge Robles, Graduate Research Assistant

Mr. Meininger completed his Master of Science degree under this program in September, 1990. He is now employed at the AFLC at McClellan Air Force Base.

In July, Dr. Koji Tanaka of Tokyo University will join our group as a visiting research scientist. Dr. Tanaka will provide valuable assistance in the area of high resolution TEM, which is needed in order to fully characterize the materials that we have developed and are testing.

7. RECENT AFOSR SUPPORTED PUBLICATIONS AND PRESENTATIONS

1. CAVITATION AND FRACTURE OF IN9052 AND IN90211 MECHANICALLY ALLOYED ALUMINUM AT HIGH TEMPERATURE,
T.R. Bieler, L.K. Sadilek, S.F. Meagher and A.K. Mukherjee, to be published in <u>Hot Deformation of Aluminum Alloys</u>, TMS-AIME, Warrendale, PA.

2. DEFORMATION MECHANISMS IN TWO MECHANICALLY ALLOYED ALUMINUM ALLOYS AT HIGH HOMOLOGOUS TEMPERATURES,
T.R. Bieler, S.F Meagher, J.A. Diegel and A.K. Mukherjee, to be published in <u>Hot Deformation of Aluminum Alloys</u>, TMS-AIME, Warrendale, PA.

3. SUPERPLASTICITY IN METALS, CERAMICS AND INTERMETALLICS,
A.K. Mukherjee, to be published in Volume 6 in <u>Plastic Deformation and Fracture of Materials</u>, H. Mughrabi, volume editor, in the series "Materials Science and Technology" by VCH Verlagsgesellshaft mbH, Germany.

4. CREEP BEHAVIOR OF Nb-1%Zr AT INTERMEDIATE TEMPERATURES,
M.J. Davidson, M.A. Biberger and A.K. Mukherjee, to be published in "Proceedings of the Fifth International Conference on Creep", ASM International, Materials Park, OH.

5. THE ROLE OF MIXED CLIMB PROCESS OF DISLOCATION CREEP OF PARTICLE HARDENED MATRIX,
A.K. Mukherjee, Annual Meeting, Japan Institute of Metals, Sendia, Japan, 1990.

6. CREEP AND AGING RESPONSE OF A RAPIDLY SOLIDIFIED Al-Fe-V-Si ALLOY,
R. J. Lewis and J. C. Gibeling, to be published in Scripta Metallurgica et Materialia.

7. ELEVATED TEMPERATURE CREEP OF A RAPIDLY SOLIDIFIED Al-Fe-V-Si ALLOY,
R. J. Lewis and J. C. Gibeling, presented at the TMS Annual Meeting, Las Vegas, NV, March 1989.

8. CREEP AND AGING RESPONSE OF A RAPIDLY SOLIDIFIED Al-Fe-V-Si ALLOY,
R. J. Lewis and J. C. Gibeling, presented at AeroMat '90, Long Beach, CA, May, 1990.

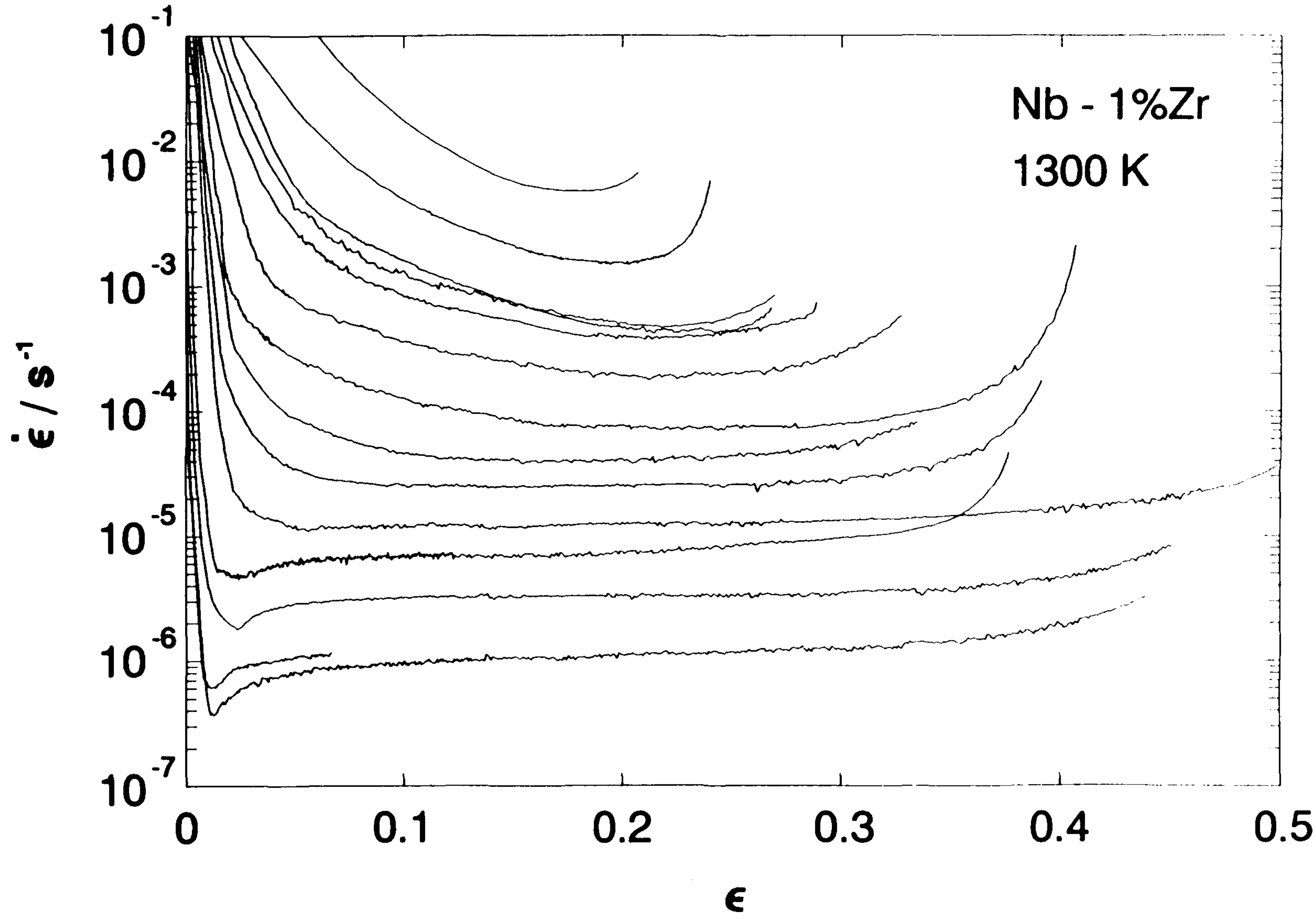

Fig. 1 Creep rate $\dot{\epsilon}$ as a function of strain ϵ for Nb-1Zr tested at 1300K at various stresses.

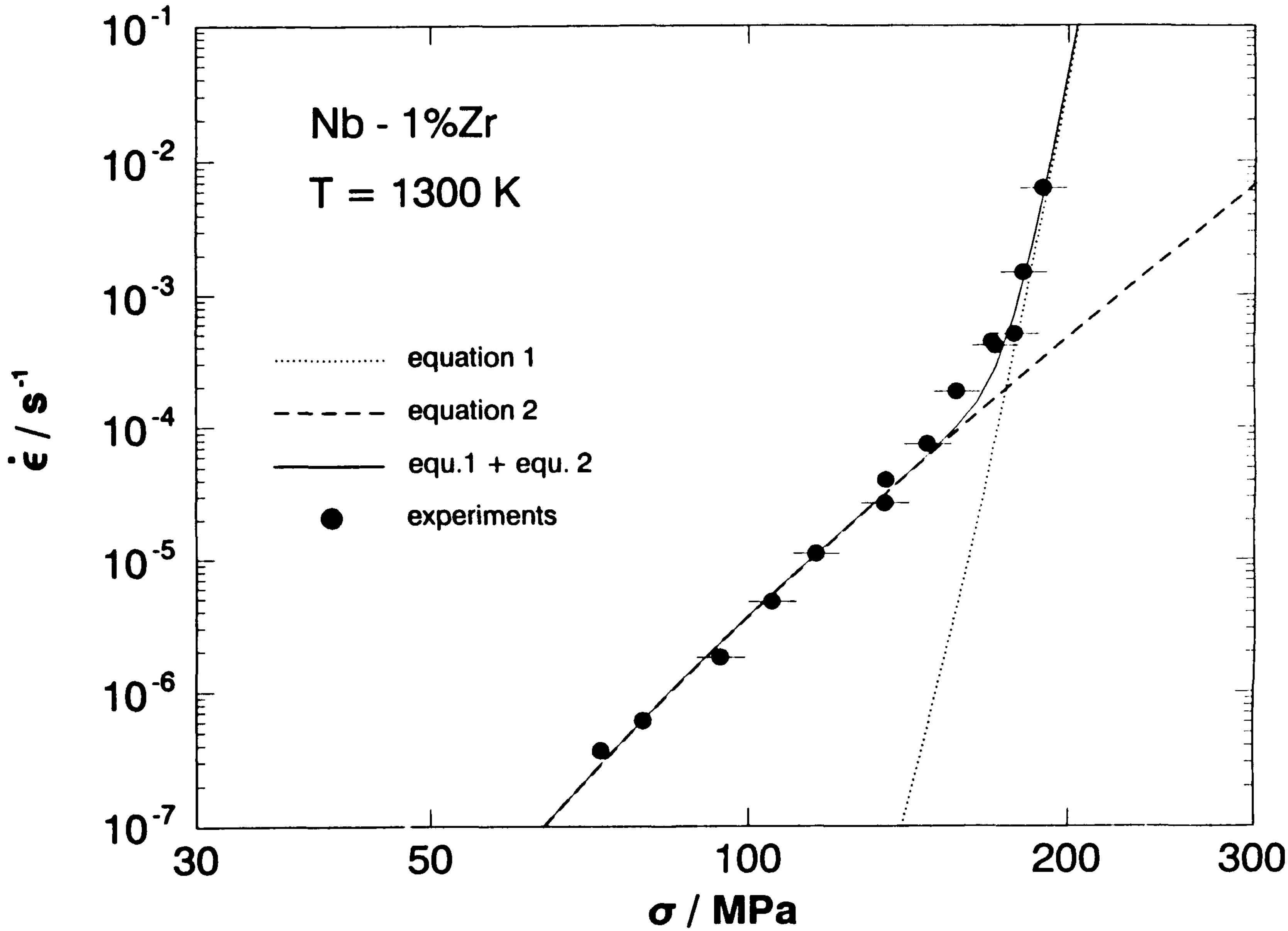

Fig. 2 Creep rate $\dot{\epsilon}$ as a function of stress σ for Nb-1Zr tested at 1300K. The dotted line (Eq. 1), Dashed line (Eq. 2) and the solid line (Eq.1 + Eq. 2) represent a model to describe the measured creep rates.

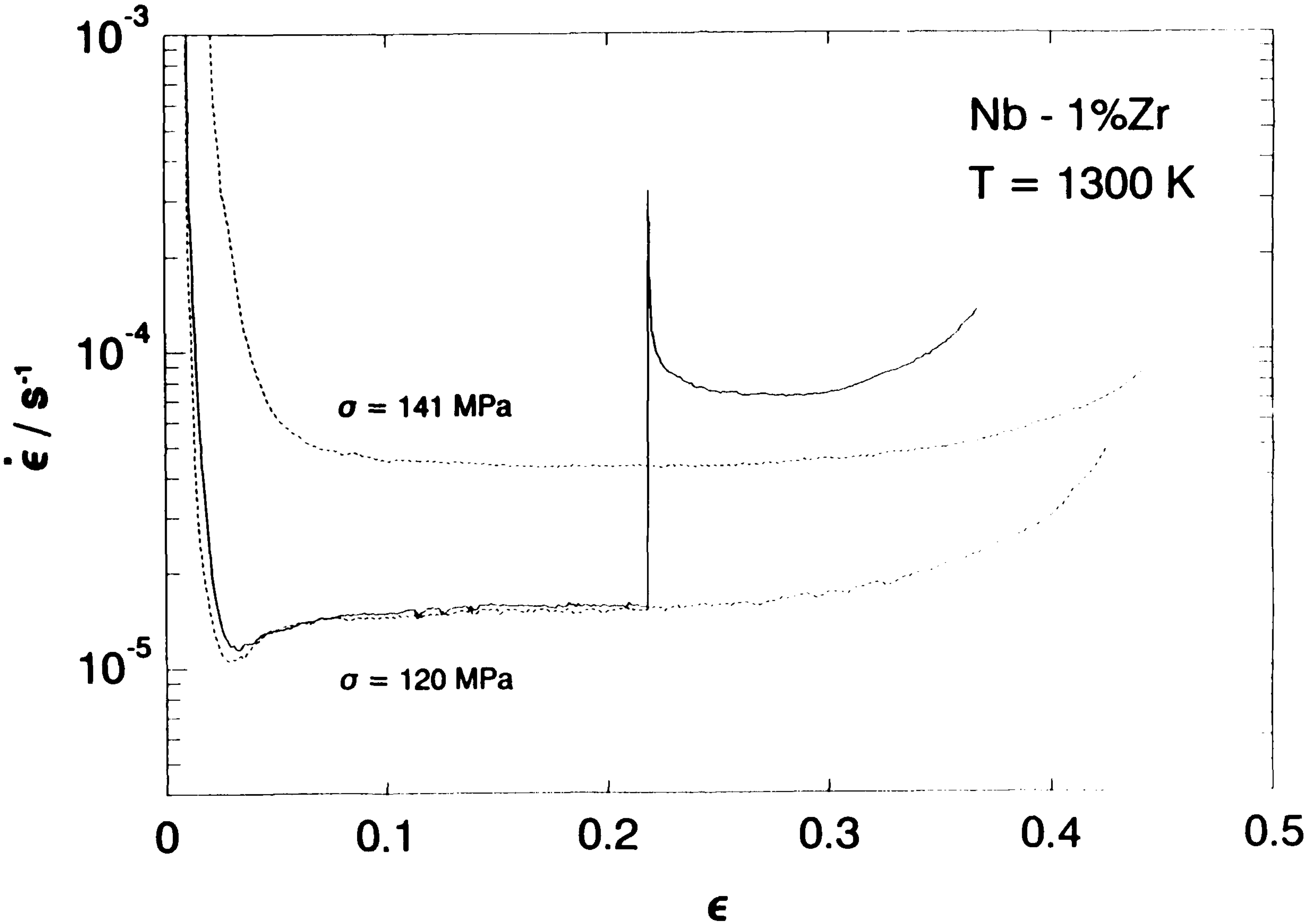

Fig. 3 Creep rate $\dot{\epsilon}$ as a function of strain ϵ for Nb-1Zr tested at 1300K. Plotted are two monotonic tests at 120 MPa and 141 Mpa (dashed lines) as a well as the result of a stress change from 120 MPa to 141 MPa (solid line).

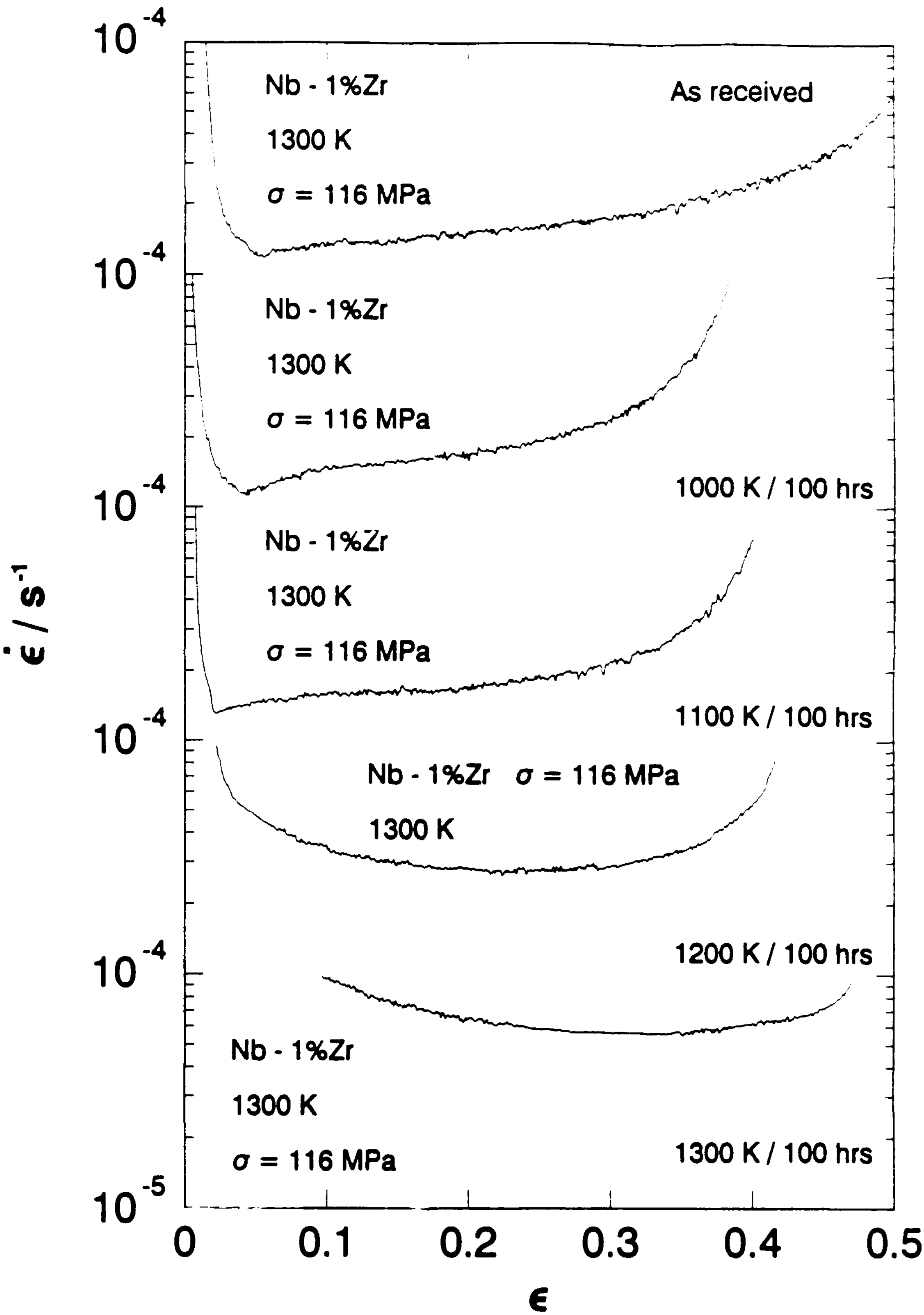

Fig. 4 Creep rate $\dot{\epsilon}$ as a function of strain ϵ for Nb-1Zr as-received and annealed in vacuum and tested at 1300K at 116 MPa.

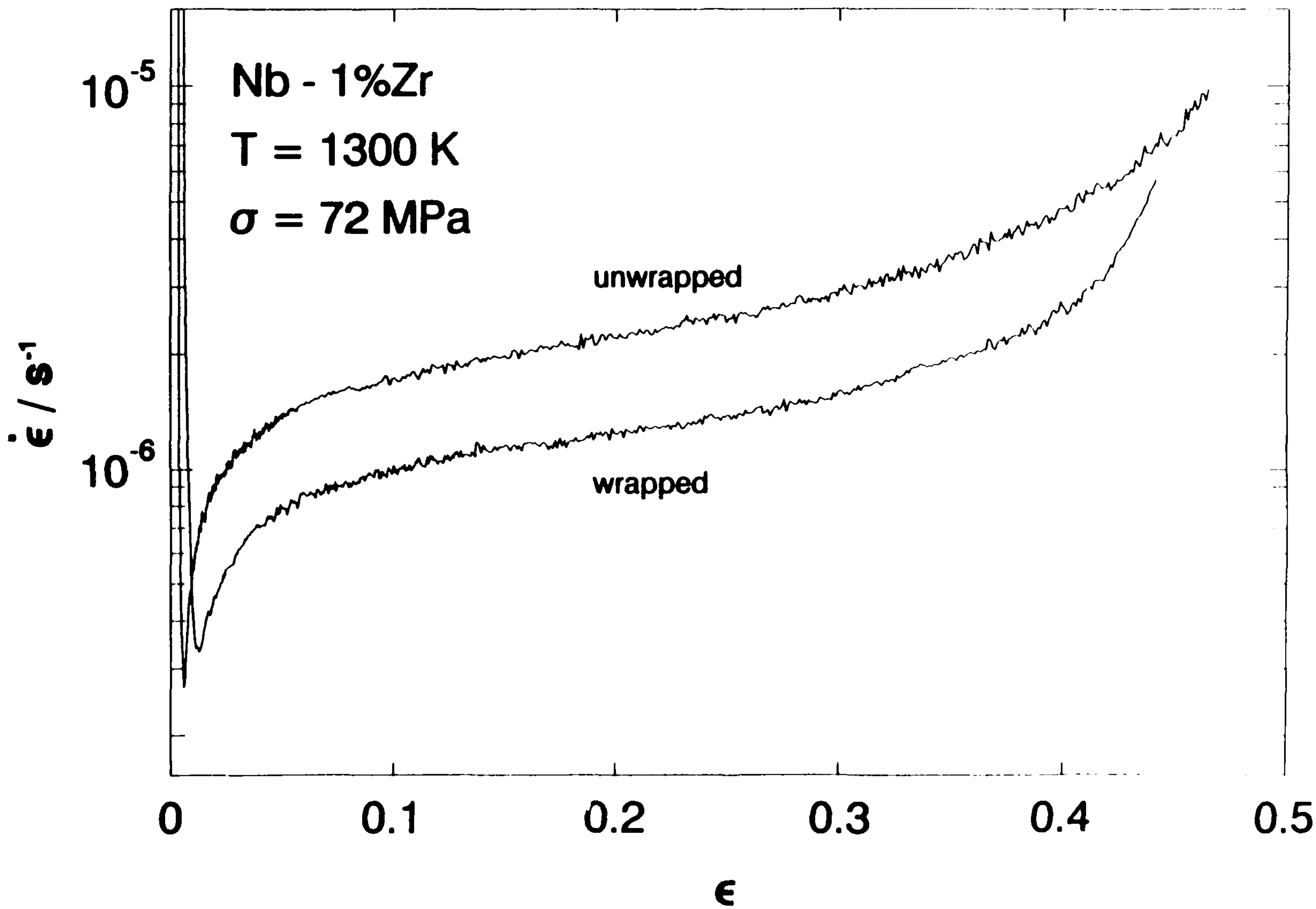

Fig. 5 Creep rate $\dot{\epsilon}$ as a function of strain ϵ for unwrapped (upper curve) and wrapped (lower curve) Nb-1Zr tested at 1300K at 72 MPa.

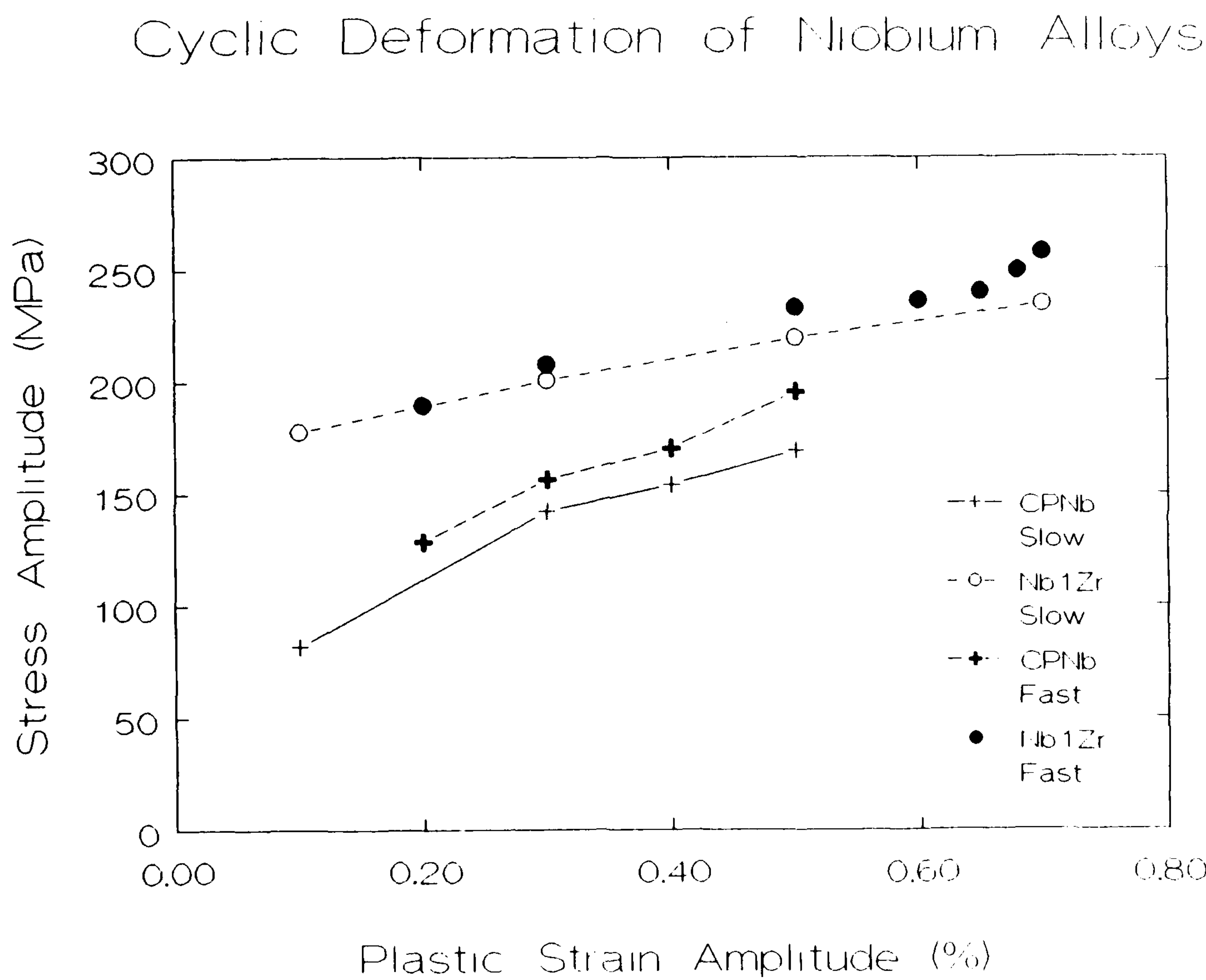

Fig. 6 Room temperature cyclic stress strain curves for commercially pure Nb and Nb-1Zr tested at high and low strain rates.

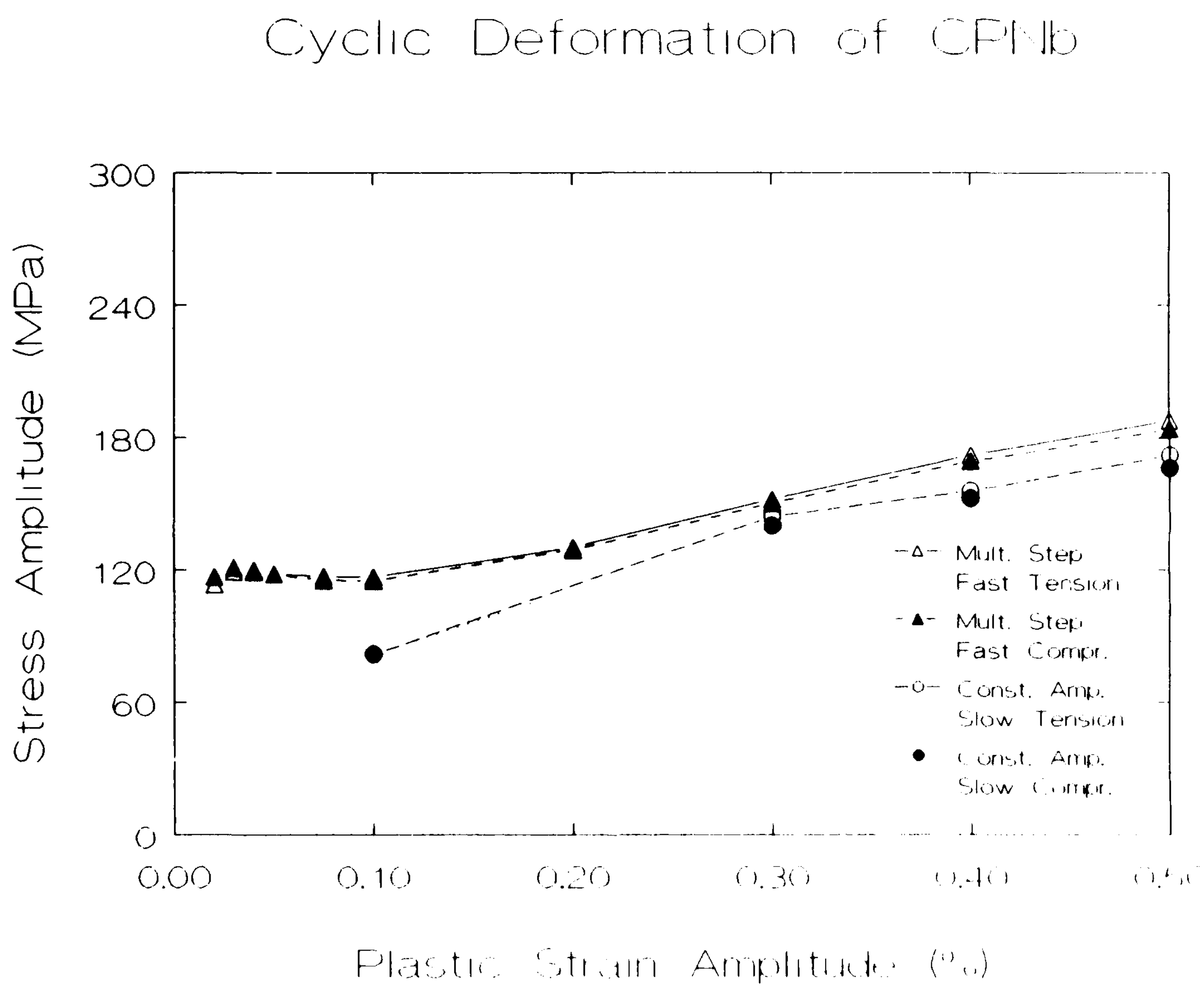

Fig. 7 Room temperature cyclic stress strain curve for commercially pure Nb showing a microplastic plateau at the fast strain rate.