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SUPERPLASTICITY IN A NICKEL SILICIDE ALLOY-MICROSTRUCTURAL AND MECHANICAL CORRELATIONS

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ABSTRACT

The superplastic properties of a nickel silicide based intermetallic alloy have been investigated as a function of strain, strain-rate, and temperature. The evolution of the microstructure during superplasticity, including grain growth, grain refinement, and cavitation, is reviewed. The relationship of the flow stress to strain and to strain-rate and the activation energy of the deformation process have been established. Finally, deformation mechanisms for superplasticity in the alloy at intermediate and high strain-rates are proposed.

INTRODUCTION

Superplasticity is the ability of a material to undergo extensive elongation without failure. Superplastic materials may show uniaxial tensile elongations of thousands of percent. The basic requirements for superplasticity are: 1) a fine, equiaxed, stable microstructure, 2) a temperature in excess of half the melting point of the material, 3) mobile grain boundaries to facilitate grain boundary sliding and 4) a controlled strain-rate.

Superplastic forming (SPF) takes advantage of the extensive ductility of superplastic materials to form complex, monolithic components, reducing weight and eliminating costly assembly time. The ability to form near net shape parts by SPF also minimizes machining and reduces material waste.

Advanced aircraft and power plant designs require materials for elevated temperature structural applications that are strong and lightweight. Ordered intermetallic alloys are a class of materials that generally meet these criteria. In recent years, several intermetallic alloys have shown superplasticity. It may be desirable to take advantage of the SPF potential of these materials. This requires an understanding of their microstructural characteristics and their correlation to mechanical properties. This paper reviews these items for an Ni_3Si-based intermetallic alloy.

EXPERIMENTAL PROCEDURE

The nominal composition of the material used for this study was Ni - 9 wt% Si - 3.1 wt% V - 4 wt% Mo. The V and the Mo were added to the binary Ni_3Si to promote superplasticity by stabilizing the beta phase. The thermomechanical processing of the material included hot forging of the initial cast ingot, hot rolling to the final thickness, and several anneal steps.

Tensile specimens were prepared from the material by electrical discharge machining with the tensile axis parallel to the rolling direction. The specimens were tested using a digital controlled Instron 4505 tensile machine, interfaced with a data acquisition system. Tests were performed in a purified Argon atmosphere in a radiant heat furnace. Change of strain-rate ($\dot{\varepsilon}$) tests and constant true strain-rate tests were conducted. A temperature range of 1298 K to 1363 K and $\dot{\varepsilon}$ from $6*10^{-5}$ to 10^{-1} s^{-1} were investigated.

The material was examined using optical metallography, scanning electron microscopy, and transmission electron microscopy. An electron microprobe was used to get chemical compositions. Grain sizes were determined by a linear intercept method and by a Quantimet Image Analyzer. Cavitation data was collected on a Ziess Image Analysis system.

RESULTS AND DISCUSSION

Microstructural Characteristics

The three dimensional microstructure of the Ni_3Si alloy, as-received, is shown in Figure 1. The material shows anisotropy and a large distribution of grain sizes. Ideal superplastic microstructures contain fine, equiaxed grains, uniform in size.

Three phases are present in the Ni_3Si(V,Mo). The dark grains are β phase with an $L1_2$ type cubic structure. The light grains are comprised of α - Ni solid solution phase and β phase (designated $(\alpha+\beta)$ from here on). The cubic ordered β phase is present as dispersions in the disordered α matrix. The third phase is randomly distributed in the microstructure and appears as dispersions, 1 to 2 um in diameter. It is approximately 30 at% Mo. There is no apparent change in the size, morphology or volume fraction of this phase with deformation, indicating it is insignificant to the superplasticity of the Ni_3Si alloy.

The average size of the $(a+\beta)$ grains in the as-received material is 14 um. (Grain sizes are expressed as an average linear intercept value measured in the plane defined by the longitudinal and long transverse axes.) The average β phase grain size is 3 um. Both phases show a wide distribution in grain sizes.

During superplastic deformation, refinement is observed in the $(\alpha+\beta)$ grains while strain-enhanced growth occurs in the β phase grains. Figure 2 shows strain-enhanced grain growth/refinement as a function of local strain (expressed as a reduction in area). Data is presented for three strain-rates at a constant temperature. A reduction in $(\alpha+\beta)$ grain size with strain is apparent at all strain-rates. In the β phase, strain-enhanced grain growth increases with decreasing strain-rate. The lack of grain growth at the higher $\dot{\varepsilon}$ is consistant with the work of Caceras, et al. [1]. In a number of superplastic materials, they showed a tendency for the grain growth rate to reach a plateau at high strain-rates due to the inability of grain boundaries to migrate.

High temperature annealing (static) also induces refinement in the $(\alpha+\beta)$ grains and growth in the β phase grains. A four hour anneal at 1323K reduced the difference in the average size between the two phases as well as the size distribution in both phases. A specimen that was first annealed by this method was tested in tension. It demonstrated superior elongation, suggesting that the superplastic performance of this alloy might be improved by first annealing the material. Further research is being conducted to understand the enhanced superplasticity and its possible relation to an increase in tensile stability due to strain-enhanced grain growth, as suggested by Wilkinson [2].

Ni_3Si(V,Mo) exhibits cavitation during superplastic deformation. Cavities are located almost exclusively at $(\alpha+\beta)$ / β interfaces. It is common for cavities to be located at grain boundaries in superplastic materials due to the extent of grain boundary sliding during superplasticity. The cavities in the Ni_3Si alloy are rounded at lower strains. At higher levels of strain they grow either in a crack-like fashion or coalesce into short stringers parallel to the tensile axis. The presence of very small round cavities near the fracture tip indicates that cavities are nucleated continuously during superplasticity.

Figure 3 shows the volume % of cavities as a function of local strain, expressed as a reduction in area, for three strain-rates. For a given strain (ε), cavitation increases with decreasing strain-rates. The data also suggests that cavitation significantly increases for all strain-rates at ε = 1.4. Inspection of the micrographs indicate that this is the strain level at which cavity coalescence becomes significant.

The micrograph in Figure 4 shows the fracture tip of a specimen that was superplastically deformed to failure at 1343 K and $\dot{\varepsilon}$ = $10^{-3}s^{-1}$. The substantial cross-sectional area remaining at the fracture tip indicates that final failure in this material occurs by cavity coalescence, rather than by plastic instability. Examination of the fracture tip by SEM indicates an intergranular failure mechanism, which is consistent with cavitation at grain boundaries.

Mechanical Properties and Mechanisms

The most important mechanical characteristic of a superplastic material is its high strain-rate sensitivity of flow stress. The strain-rate sensitivity, m, provides a measure of a materials ability to resist necking. It is defined by the equation:

$$\sigma \propto \dot{\varepsilon}^m \qquad (1)$$

where σ is the flow stress. The strain-rate sensitivity varies with grain size, temperature and strain-rate. To optimize superplasticity, it is useful to determine the conditions at which m has the highest value.

The strain-rate sensitivity as a function of strain-rate and temperature may be derived from change of strain-rate tests. The results of these tests conducted at temperatures between 1323 K and 1363 K and at $\dot{\varepsilon}$ from $6*10^{-5}$ to 10^{-1} s^{-1} for the Ni_3Si alloy are presented in Figure 5. The shift of the peak m to higher strain-rates with increasing temperature is characteristic of superplastic materials. This curve shows an optimum m value of approximately 0.5 at $\dot{\varepsilon}$ = 10^{-4} to 10^{-3} s^{-1}. At 1363 K and $\dot{\varepsilon}$ = $6*10^{-4}$ s^{-1}, a tensile elongation of 665% was obtained in the Ni_3Si(V,Mo). This specimen is compared to an undeformed specimen in Figure 6.

The relationship between the flow stress and the strain-rate is also derived from these tests. A sigmoidal logarithmic σ - $\dot{\varepsilon}$ curve is characteristic of superplastic materials. Optimum superplasticity occurs in the center region, Region II. The $\sigma - \dot{\varepsilon}$ data for the Ni_3Si alloy is shown in Figure 7. The range of data obtained was limited by testing so the transitions between the three characteristic regions are not well-defined. The authors believe that Region II for $Ni_3Si(V,Mo)$ lies between $5*10^{-4}$ and 10^{-2} s^{-1}. From Figure 5, m is approximately 0.5 in Region II and in Region III, the high strain-rate region, m is approximately 0.3.

In Region III, dislocation climb creep is quoted as the rate-controlling deformation mechanism in most superplastic materials. The authors believe however, that the deformation of superplastic intermetallic alloys in Region III is controlled by the viscous glide of dislocations. The viscous motion is related to the introduction of disorder in the crystal as dislocations glide, and the subsequent reinstating of order by chemical diffusion. For further discussion the interested reader is directed to reference 1.

In Region II, grain boundary sliding accommodated by the climb of dislocations at grain boundaries is the rate-controlling mechanism. This is given by the models of Mukherjee [4] and Langdon [5] for an m value of 0.5. In Region II where strain-rates and subsequently dislocation velocities are lower, the viscous glide motion of dislocations in intermetallic alloys can keep up with the imposed strain-rate.

Figure 8 shows true stress - true strain curves for three constant strain-rate tests conducted at 1343 K. The lower two strain-rates are in Region II and the higher one in Region III. The strain hardening demonstrated by the curves in Region II is related to dynamic grain growth of the β phase. The strain softening at the higher strain-rate can be attributed to early onset of dynamic insitu grain refinement in the $(\alpha+\beta)$ phase, as illustrated in Figure 2.

The apparent activation energy (Q) of deformation was calculated from the σ - $\dot{\varepsilon}$ curves for Regions II and III in the Ni_3Si alloy. Q in Region II is 526 kJ/mol and in Region III is 495 kJ/mol. As there is no reported diffusion data for Ni_3Si, the measured activation energies cannot be related to any specific deformation mechanism at this time. Nieh [6] has reported an activation energy of 555 kJ/mol for the same superplastic Ni_3Si alloy.

CONCLUSIONS

Superplasticity in Ni_3Si(V,Mo) has been demonstrated. A maximum tensile elongation of 665% has been demonstrated at $\dot{\varepsilon} = 6*10^{-4}\ s^{-1}$ at 1363 K.

During superplastic deformation, grain refinement is observed in the grains consisting of an $(\alpha+\beta)$ phase mixture while strain-enhanced growth occurs in the β phase grains. Cavitation is observed during superplasticity, and increases with decreasing strain-rate for a given level of strain.

The activation energies in Regions II and III are 526 kJ/mol and 495 kJ/mol, respectively. At this time these values cannot be associated with any specific diffusion process. The rate-controlling deformation mechanism in this alloy is believed to be governed by the climb of dislocations at grain boundaries in Region II, and the viscous glide of dislocations in Region III.

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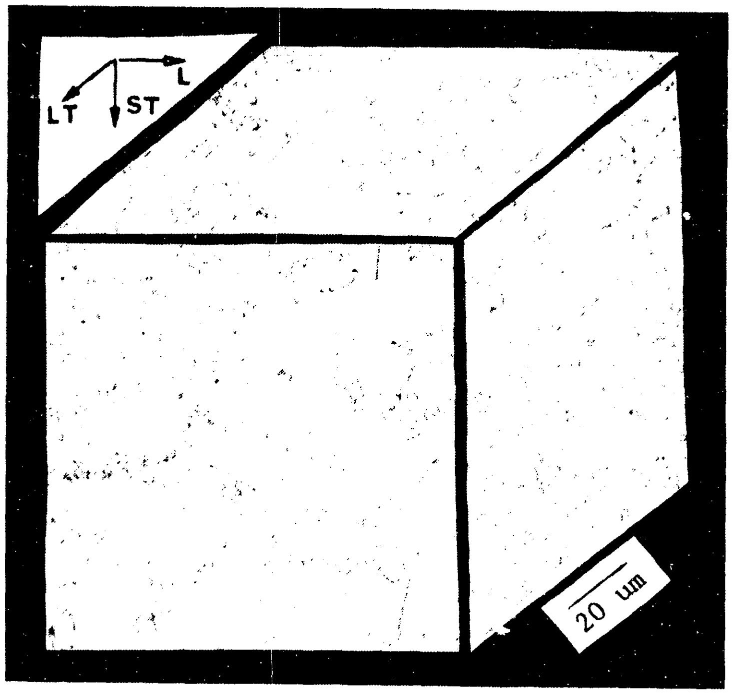

Fig 1. Microstructure of the as-received Ni_3Si(V,Mo). The dark grains are $L1_2$ cubic β phase and the light grains consist of an $(\alpha+\beta)$ phase mixture.

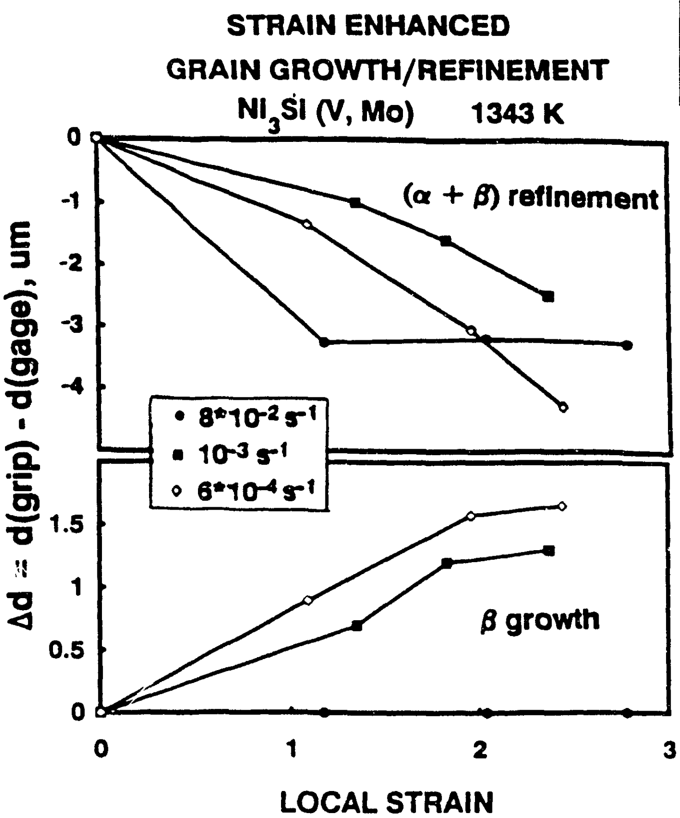

Fig 2. Strain-enhanced refinement of the $(\alpha+\beta)$ phase grains and strain-enhanced growth of the β phase grains as a function of strain-rate. The average sizes of the $(\alpha+\beta)$ and the β grains in the as-received material are 14 um and 3 um, respectively.

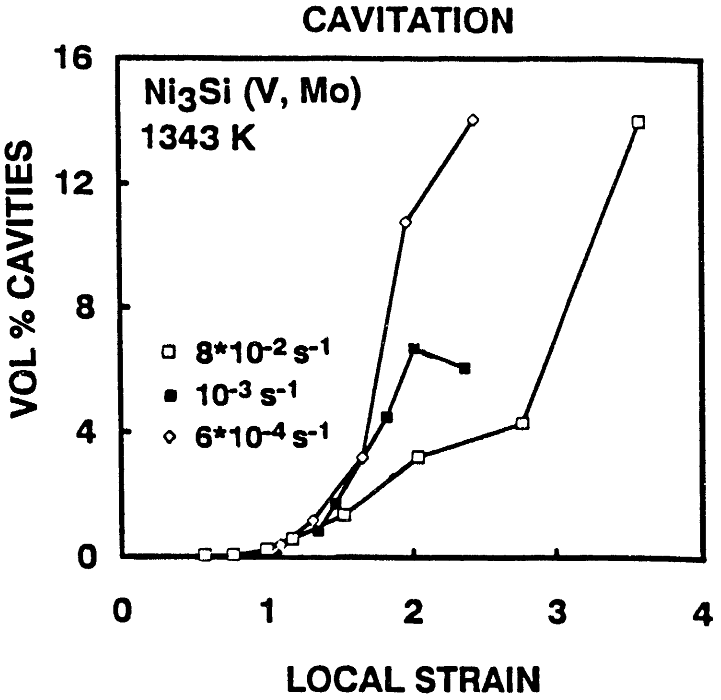

Fig 3. Volume % cavitation as a function of local strain for three strain-rates at a constant temperature.

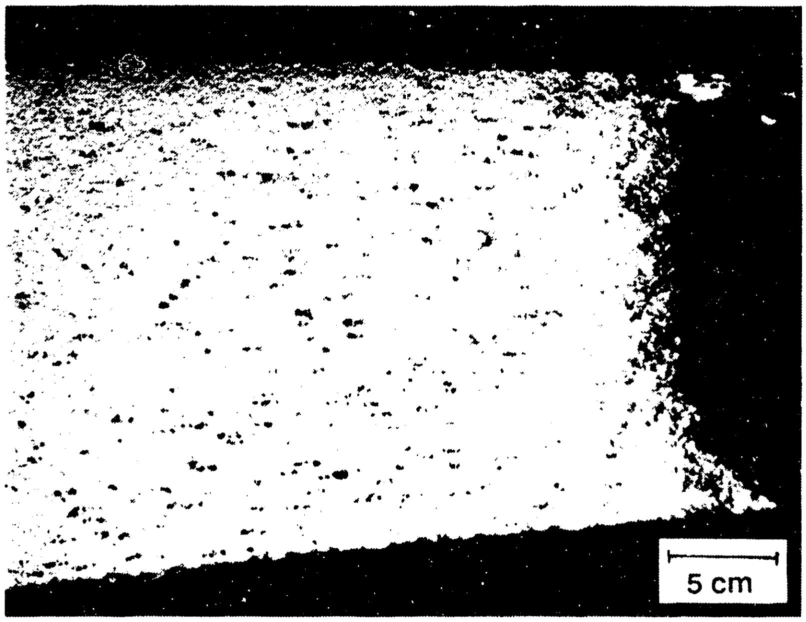

Fig 4. Cavitation shown at the fracture tip of a tensile specimen superplastically deformed to failure at 1343 K and ε = 10^{-3} s^{-1}.

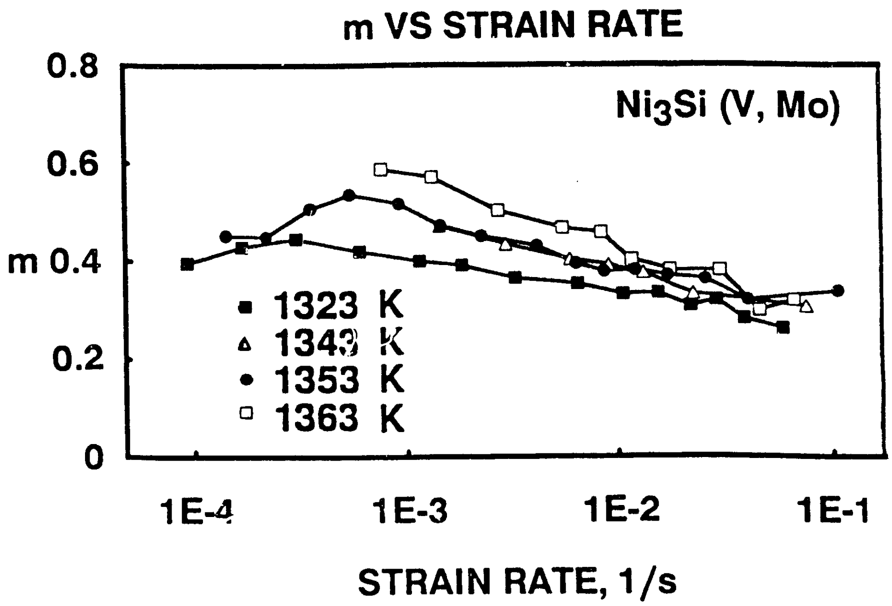

Fig 5. Strain-rate sensitivity as a function of temperature and strain-rate.

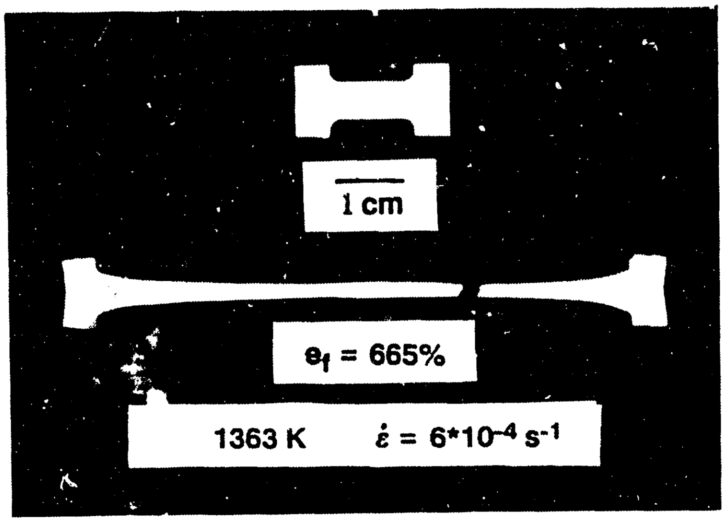

Fig 6. Comparison of an undeformed $Ni_3Si(V,Mo)$ specimen to one that has been superplastically deformed at 1363 K.

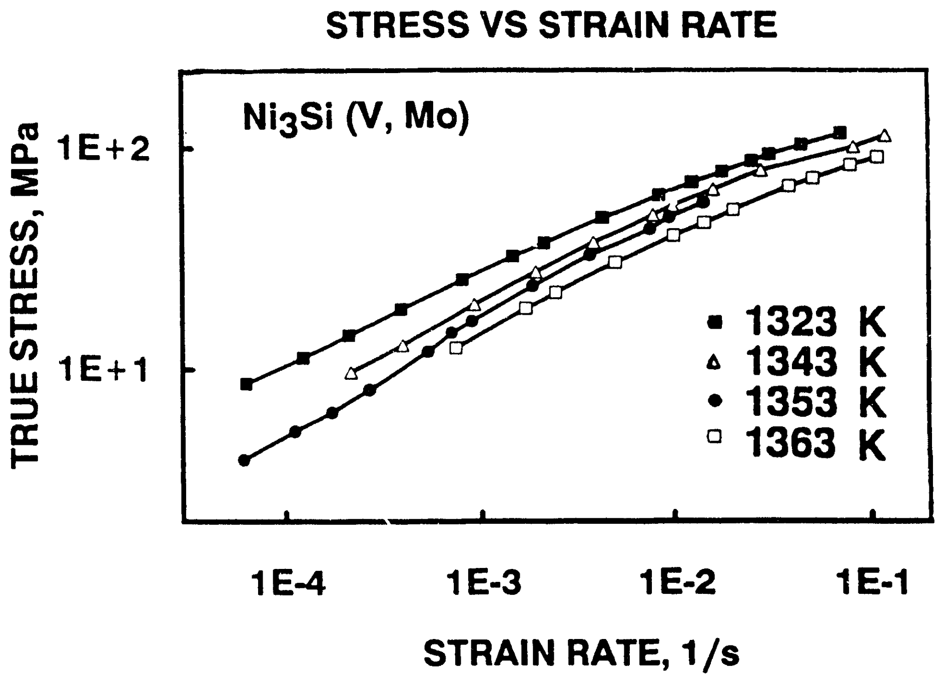

Fig 7. Relationship between the flow stress and the strain-rate as a function of temperature.

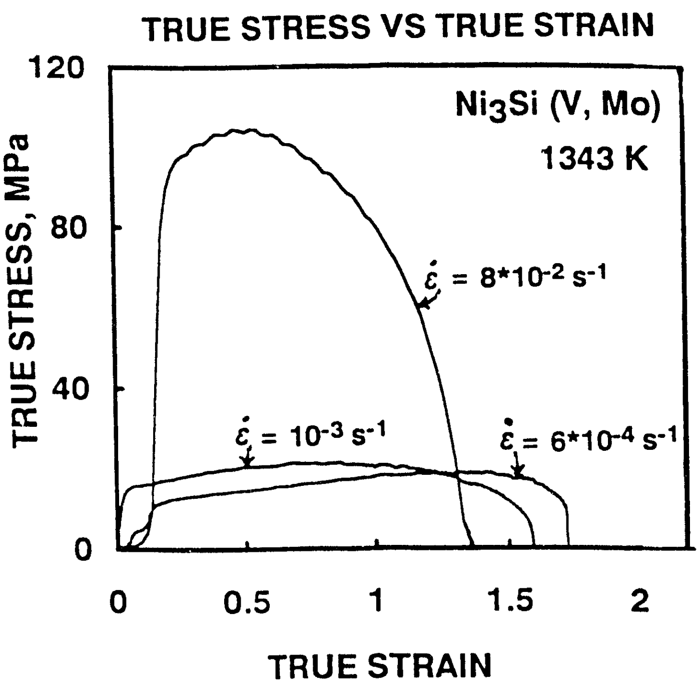

Fig 8. True stress - true strain curves for constant strain-rate tests.

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