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GRAIN BOUNDARY INTERNAL FRICTION AND RELATIONSHIP TO INTERGRANULAR FRACTURE

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Abstract

Creep rate, stress rupture and fracture mode have been measured on Ni-20 W/O Cr alloys containing 0 and 180 at. ppm Ce. Grain boundary viscosity and anelastic relaxation phenomena have been determined by high temperature internal friction measurements and results correlated to fracture behavior.

Introduction

The phenomena of high temperature intergranular fracture with its associated very low alloy ductility is a complex process which include cavity nucleation, cavity growth, interlinkage followed by final fracture [1,2]. It is now well established that localized high stress concentration in excess of applied stress is required for the nucleation of cavities [2]. This high stress can be generated by, for instance, grain boundary sliding. In addition, grain boundary segregation is of great importance since it can reduce grain boundary cohesion and surface free energy which would in turn reduce the critical stress required for cavity formation. For instance, it is known that trace amounts of S promote intergranular fracture [3], while others such as Ce [4] or B [5] restore high alloy ductility. Finally, fracture path is also determined by test conditions and in general intergranular fracture is observed predominantly at high temperatures and for low strain rate tests. This behavior is observed because at low temperatures and high strain rates, grain boundaries are essentially immobile and therefore cannot generate the required localized high stresses. An upper bound strain rate, above which cavitation does not occur, has been obtained by Crossman and Ashby [6] by considering the balance between stress concentration generated by grain boundary sliding and stress relaxation produced by dislocation creep. This upper bound or transition strain rate (TSTR) is given by:

$$\dot{\epsilon}_{TSTR} = \left\{ \frac{1}{A} \left[\frac{\mu \delta}{\eta_{gb} d} \right]^n \right\}^{1/n-1} \quad (1)$$

where η_{gb} denotes the grain boundary sliding viscosity, δ the grain boundary thickness, d the grain size, n and A are the stress exponent and parameter of the power-law creep relation:

$$\dot{\epsilon} = A \left(\frac{\sigma}{\mu} \right)^n \quad (2)$$

with

$$A = A_0 e^{-Q_c/RT} \quad (3)$$

where Q_c denoted the creep activation energy. All the parameters involved in eq. 1 are known or can be determined experimentally. In particular, grain boundary sliding viscosities can be measured directly by the internal friction method [7,8].

This paper presents the results of an investigation on the upper bound strain rate for intergranular fracture in a Ni-20 Cr alloy. First, the formalism of internal friction measurements is presented which is followed by a dislocation glide description for grain boundary relaxation time. Internal friction and creep data are then used to model the predicted transition strain rate which is then compared to experimental data. Finally, the possible roles of minor Ce additions on the observed suppression of intergranular fracture are discussed in light of internal friction results.

Internal Friction Analysis

The internal friction technique, although indirect, is unique for the study of grain boundaries and has been used successfully to determine grain boundary sliding kinetics [7,8] and grain boundary segregation phenomena [9]. Whenever grain boundaries can move freely, viscous sliding occurs leading to an exponential increase in high temperature background. If sliding is impeded by pinning points, anelastic relaxation will occur leading to a peak in internal friction. This relaxation process is characterized by a relaxation time τ which is expressed as

$$\tau = \tau_0 e^{Q/RT} \quad (4)$$

A detailed basic mechanism for grain boundary internal friction has not yet been clearly established, although models based on grain boundary sliding by diffusion have received most attention [7,8]. Since grain boundaries are composed of discrete dislocations, grain boundary sliding may be described also by the climb process of these dislocations. In this case, the relaxation time is given by [10]:

$$\tau = \frac{\lambda}{v_c} = \frac{\lambda b^2}{M \sigma \Omega} \quad (5)$$

where v_c is the average dislocation climb velocity, λ the climb distance, σ the shear stress, Ω the atomic volume, b the dislocation burgers vector and M the mobility. Two limiting cases can be considered for the dislocation mobility [10]. For a grain boundary free of segregated impurities the intrinsic dislocation mobility is given by

$$M = \frac{D_{gb} b}{kT} \quad (6)$$

where D_{gb} is the grain boundary diffusivity. For an alloy containing impurity atoms, the dislocation mobility will be limited by the drag caused by segregated impurities. In this case the dislocation mobility decreases to [10]

$$M = \frac{D_s \Omega}{b^2 kT \beta C_0} \quad (7)$$

where D_s is the solute diffusivity, β the grain boundary enrichment factor and C_0 the impurity bulk concentration.

Finally, grain boundary viscosity η_{gb} is related to relaxation time via the following equation [7,8]

$$\eta_{gb} = \frac{E \delta}{\ell} \tau = \frac{E \delta}{\ell} \tau_0 e^{Q/RT} \quad (8)$$

where E is the modulus of elasticity, δ the grain boundary thickness and ℓ the

length of sliding grain boundaries, i.e. distance between pinning points. In most studies, an equiaxed grain structure is considered and ℓ is taken as the average grain size d .

Experimental Procedures

Tensile tests, creep tests and internal friction measurements were performed on two Ni-20 W% Cr alloys containing 0 and 180 at. ppm Ce. The alloys were prepared by vacuum induction melting at Special Metals Corporation, followed by casting and hot rolling. Similar heat treatments were performed on all alloys consisting of a vacuum anneal at 1100°C in order to obtain similar grain sizes ($\sim 80\mu\text{m}$) followed by aging for 24 hours at 800°C.

Internal friction measurements were performed at low frequency (0.5 - 3 Hz) in a classical torsion pendulum and data deduced from the wave form analysis of the free decay signal [11]. Strain amplitude was 5×10^{-6} and heating and cooling rates 2°K/min.

Stress exponents and creep activation energies were obtained using a constant load creep tester. All data were analyzed by converting results to true stress data by measuring true strain at minimum strain rate [12]. Ductilities, fracture modes and transition strain rate data were obtained by combined creep and tensile testing using a constant crosshead velocity machine [4].

Results and Discussion

A typical internal friction spectrum Q^{-1} measured on a function of temperature is depicted in Fig. 1 for the alloy without Ce. The spectrum reveals a peak which is superposed to an exponential background. The activation energy for the peak Q , and relaxation time, τ_0 , have been determined for both alloys by measuring the shift in peak temperature T_p as a function of frequency in an Arrhenius type plot. These results are summarized in Table I. By inspection of this Table it can be seen that trace additions of Ce decreases the activation energy from a high value of 370 kJ/mole to a lower value of 200 kJ/mole and decreases the peak temperature measured at 1 Hz. In the literature it is generally observed [14] that small additions of both substitutional and interstitial atoms increase the peak activation energy with the appearance of a new peak at large concentrations. In addition, if the rate controlling process for grain boundary internal friction is the glide and climb process of grain boundary dislocations, then the activation energy increases from grain boundary diffusivity to that of impurity self diffusivity. This result seems to indicate that additions of Ce decreases the impurity content of the alloy and their segregation to grain boundaries. The precise grain boundary chemistry is not known for these alloys but a reduction in bulk O and S content has been measured upon Ce additions [13]. In view of the strong tendency of O and S to segregate at grain boundaries, (i.e. large β values) a similar decrease in grain boundary chemistry is expected.

Table I Internal friction peak temperature, T_p , measured at 1 Hz, relaxation time τ_0 , activation energy Q and relaxation strength Δ as a function of Ce concentration. Also shown are the grain boundary viscosities η evaluated at 700°C.

Ce Concentration [at. ppm]	T_p [°C]	τ_0 [s]	Q [kJ/mole]	Δ [$\times 10^{-3}$]	η [Ns/m ²]
0	710	1.9×10^{-21}	370	2.3	2.7×10^5
180	685	2.6×10^{-12}	200	2.2	1.2×10^5

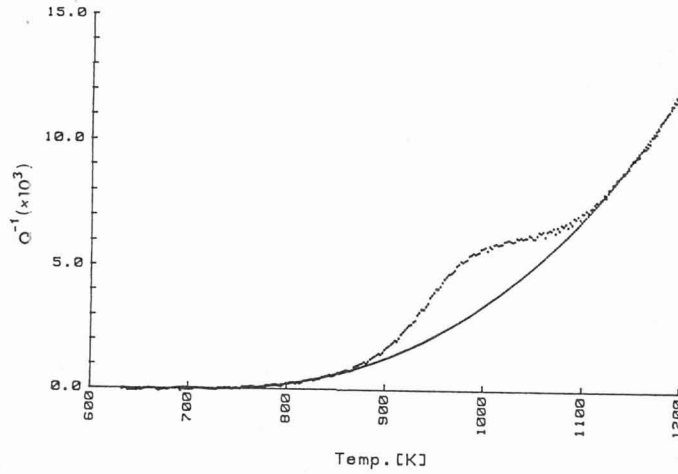


Figure 1 Internal friction spectrum Q^{-1} versus temperature for Ni-20Cr alloy containing 0 ppm Ce. The background is fitted with a single exponential.

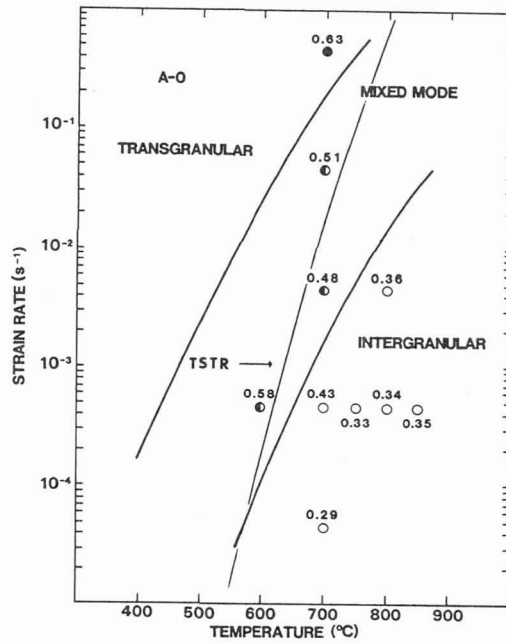


Figure 2 Fracture behavior as a function of strain rate and temperature for Ni-20Cr alloy without Ce. Filled, open and half open symbols represent in turn, transgranular, intergranular and mixed fracture modes. Numbers represent elongation at fracture. The TSTR line is the theoretical transition strain rate.

Table II Stress exponent (n), prestress exponent coefficients (A) and A_0 , and creep activation energy (Q_c) as a function of Ce concentration.

Ce Concentration [at. ppm]	n	A [1/s]	A_0 [1/s]	Q_c [kJ/mole]
0	5.2	5.5×10^{10}	5.8×10^{26}	295
119	5.2	1.9×10^{10}	8.6×10^{22}	345

In order to determine the transition strain rate, creep stress exponents and creep activation energies have been measured for various Ce containing alloys [12]. The results are summarized in Table II. Contrary to grain boundary relaxation data, creep activation energy increases with Ce content. However, Ce has little effect on the stress exponent n which is equal to 5.2 for all compositions. This increase in activation energy was attributed to an increase in bulk diffusivity [12].

Finally, alloy ductility and fracture mode have been measured as a function of test strain rate and temperature [4]. The results for the alloy without Ce are represented in Fig. 2 in a temperature-strain rate-fracture mode graph. The experimental data can be divided into three broad categories according to their fracture path. At high test temperatures and low strain-rates the fracture mode is intergranular with an associated low ductility. At low temperatures and high stresses, the fracture mode is transgranular with a corresponding high ductility. In between these two broad categories, mixed mode of fracture is observed. A drastically different picture is obtained for the alloy containing 180 at. ppm Ce. This alloy remains ductile for all test conditions and fracture mode remain transgranular.

These experimental results can now be compared with theoretical predictions given by eq. 1. The calculated TSTR values drawn in Fig. 2 show a reasonable agreement between theory and experimental results, and eq. 1 can therefore be used to estimate the ductile-brittle transition in alloys which are susceptible to intergranular fracture.

For Ce containing alloys intergranular fracture is not observed even at the lowest creep strain rates of $\dot{\epsilon} = 10^{-7} \text{ s}^{-1}$. This result indicates that either much larger stresses are required for the formation of cavities and/or that stresses can be relieved much more readily. Furthermore, a pronounced internal friction hysteresis between heating and cooling cycles was measured for the 180 ppm Ce alloy [15]. This hysteresis which was not observed for the alloy without Ce was attributed to a destabilization of the grain boundary pinning points at high temperature. This in turn could minimize stress concentration points and therefore reduce the nucleation rate of cavities and restore high temperature ductility.

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