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이차 재결정화된 기계적 합금화 ODS NiAl의 creep threshold stress에 관한 고찰

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On The Creep Threshold Stress in Secondary Recrystallized ODS MA NiAl

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ABSTRACT NiAl based ODS (Oxide Dispersion Strengthened) intermetallic alloys have been produced by mechanical alloying (MA) process and consolidated by hot extrusion. Subsequent thermomechanical treatments have been applied to induce secondary recrystallization in an attempt to improve creep resistance in this material. The creep behavior of secondary recrystallized MA NiAl has been investigated and compared with those of as-extruded condition. Minimum creep rate were shown to be approximately two orders of magnitude lower than that in as-extruded condition. The improvement in creep resistance is believed due to the grain coarsening, restricting of dispersoid coarsening as well as increase in grain aspect ratio. Creep threshold stress behavior, below which no measurable creep rate can be detected, has been discussed on the basis of particle-dislocation interaction theory. The threshold stress becomes negligible after secondary recrystallization in MA NiAl, presumably due to dispersoid coarsening and a decrease in grain boundary area during secondary recrystallization.

1. Introduction

The B_2 structure nickel aluminide(NiAl) offers potential advantages over current superalloys for use in high temperature structural applications. These advantages include high melting temperature, low density, excellent oxidation resistance and high thermal conductivity.[1] However, cast, polycrystalline NiAl suffers from poor ambient temperature ductility and poor creep resistance at intended service temperature, which indicates that the use of monolithic material is improbable. In an effort to address these problems an approach has been to use mechanical alloying (MA) followed by hot extrusion to produce dispersion strengthened NiAl based materials. The technique allowed us to produce quality powders, when consolidated, have high strength at both ambient and elevated temperatures and good compression ductility.[2,3]

The high temperature strength of ODS materials can be further improved by producing a highly elongated or fibrous grain microstructure aligned parallel to the stress axis, which reduces grain boundary sliding and minimizes transverse rupture by control of cavitation on transverse boundaries.[4] Such structures can be produced by secondary recrystallization mechanism[5] and we have therefore undertaken thermomechanical treatments to promote secondary recrystallization(SRx) in MA NiAl to further improve its creep resistance. Here, SRx represents a mechanism that can give a pronounced increase in grain size and, because it develops very rapidly, can give coarse grain structure without concurrent dispersoid coarsening.[5,6]

Recently, we examined creep behavior of as-consolidated and grain coarsened ODS MA NiAl, and demonstrated its potential for high temperature structural applications.[3,7] Minimum creep rates in as-consolidated MA NiAl were on average three orders of magnitude lower than that in their cast counterparts.[3] Improved creep resistance of MA NiAl was found to result, above all, from the presence of dispersoids. In particular, it was found that creep in the MA NiAl materials is controlled by the climb of dislocations over the dispersoids.[7] Creep resistance of SRxed MA NiAl was shown to be superior to that of as-extruded condition.[7] Improved creep resistance of SRxed MA NiAl was found to result from increase in grain size and finely remained dispersoid size as well as increase in grain aspect ratio.[7] It has also been reported that the stress exponent of creep in SRxed condition is in the normal range for the (climb controlled) dislocation creep regime though grain boundary sliding or diffusional mechanism is also accommodated in SRxed MA NiAl.[7] Thus, it is necessary to investigate dislocation creep mechanism in order to rationalize the effect of secondary recrystallization in MA NiAl.

Virtually all modern theories for dislocation creep in metals and alloys assume that dislocation motion is opposed by back stress which results from interaction between dislocations and particles in the creep condition and the creep rate is determined by an effective stress, equal to the difference between an applied stress and a back stress.[8] Here the back stress is frequently termed as a threshold stress below which creep rate can not be detectible and which must be overcome in order for dislocations to pass particles.[9] Thus, the threshold stress can be expected to change in SRxed condition due to the dispersoid coarsening.

In this study, threshold stress behavior in SRxed MA NiAl is investigated and compared with that in as-extruded condition.

2. Experimental Procedure

2.1. Materials and Creep Test

The dispersion strengthened NiAl powders are prepared by high energy ball milling of a mixture of elemental Al and Ni powders using a modified Szegvari attrition mill in an argon atmosphere. The powder was sieved to -325 mesh after milling for 70 hrs, and consolidated by hot extrusion. Hot extruded (EX) bar was produced at 1400 K with an extrusion ratio of 16 : 1 after degassing in vacuum at 800 ℃. The actual chemical composition of the alloy was (at%): 47.20 Ni, 49.34Al, 0.04C, 2.6O, 0.064N, 0.02H. The MA NiAl is an oxide (Al_2O_3) dispersion strengthened, equiaxed grain material with an average grain size of about 0.5 μm.[2]

Thermomechanical treatments utilizing prestrain and isothermal annealing were carried out to induce SRx. The prestrain was introduced by uniaxial compression to various strains in a universal testing machine. The prestrained EX specimens were isothermally annealed above T_{SRx} for one hour, as specified previously.[5]

Compressive creep tests were performed utilizing a modified Satec M-3 creep testing machine. Constant load were applied ranging from 40 to 180 MPa at 800 ℃, 850 ℃ and 900 ℃. The creep strain curve as a function of time at a given temperature and stress was obtained. Steady state creep rate ($\dot{\varepsilon}_s$) was calculated using a computerized linear regression method applied to the data in the time interval between 15 and 20 hours. Then an Arrhenius plot of $ln\ \dot{\varepsilon}_s$ versus 1/T was made to obtain the apparent activation energy (Q_{app}) for creep. A plot of $ln\ \dot{\varepsilon}_s$ versus $ln\ \sigma$ for a given microstructure was also made in order to obtain the stress exponent (n).

2.2. Determination of the Threshold Stress

In order to estimate the threshold stress of ODS MA NiAl, a well established procedure, summarized by Purushothaman and Tien,[9] was followed. The procedure requires the normalization of stresses by elastic modulus and assumes that the creep rate is determined by a net stress, equal to the difference between an applied stress and the threshold stress. As a result, the conventional power law creep equation give by:

$$\dot{\varepsilon} = A' \sigma^n \exp\left(-\frac{Q_{app}}{RT}\right) \qquad (1)$$

can be rewritten as:

$$\dot{\varepsilon} = A\left[\frac{\sigma_A - \sigma_{th}}{E(T)}\right]^{n_o} \exp\left(-\frac{Q_c}{RT}\right) \quad (2)$$

where σ_A is the applied stress, Q_{app} and Q_c are apparent and true activation energies for creep, respectively, n and n_o are apparent and true stress exponents, E(T) is elastic modulus, and A and A' are constants.

The threshold stress can now be estimated from the linear plot of the normalized strain rate against the normalized stress:

$$\left[\frac{\dot{\varepsilon}}{A\exp\left(-\frac{Q_c}{RT}\right)}\right]^{\frac{1}{n_o}} \text{ vs } \frac{\sigma_A}{E(T)} \quad (3)$$

extrapolated to the zero value of normalized stress.

To construct the plot, the values of n_o, Q_c and A in the equation had to be determined. First, the true stress exponent n_o was assumed to be equal to the apparent stress exponent. Then, the true activation energy for creep was calculated from:

$$Q_c = Q_{app} + \frac{nRT^2}{E(T)} \cdot \left(\frac{\partial E}{\partial T}\right) \quad (4)$$

Constant A was determined from the actual data (two different stresses and two different strain rates at constant temperature were considered):

$$\dot{\varepsilon}_1^{\frac{1}{n_o}} - \dot{\varepsilon}_2^{\frac{1}{n_o}} = A^{\frac{1}{n_o}} \frac{1}{E(T)}(\sigma_1 - \sigma_2)\exp\left(-\frac{Q_c}{n_o RT}\right) \quad (5)$$

Finally, the following elastic modulus/temperature relationship was used[10]:

$$E = 195.055 - 0.0349T(GPa) \quad (6)$$

The determination of the threshold stress using the graphical analysis enabled us to verify whether the assumption that $n \simeq n_o$ was justified. In as-extruded MA NiAl, the true and apparent activation energies are for all practical purposes identical and within the range of the activation energies determined in diffusion experiments.[3] As a result, the power law creep equation with the effective stress normalized by elastic modulus can be rewritten as:

$$\dot{\varepsilon} = A'\left[\frac{\sigma_A - \sigma_{th}}{E(T)}\right]^{n_o} \exp\left(-\frac{Q_c}{RT}\right) \quad (7)$$

or:

$$\frac{\dot{\varepsilon}}{D} = B\left[\frac{\sigma_A - \sigma_{th}}{E(T)}\right]^{n_o} \quad (8)$$

where D represents a diffusion coeffiecient expressed as $D=D_o\ \exp(-Q_c/RT)$. The true stress exponent n_o can now be determined from the master plot of diffusion compensated creep rate as a function of modulus corrected effective stress.

3. Results and Discussion

3.1. Materials

The primary microstructures after consolidation of MA NiAl are typically fine grained with a grain size less than 1 μm, and contain a fine distribution of Al_2O_3 dispersoids in the range of 10~100 nm.[2] A substantially SRxed microstructure was produced in the material homogeneously prestrained about 5% followed by isothermal annealing at 1300 ℃ for 30 min. Here, it was possible to have crack free prestrained specimens by compression, since MA NiAl showed significantly improved compressive ductility at room temperature due to additional slip system operations, though it was still shown to have near zero tensile ductility as in other intermetallics.[2,5] It is shown that dispersoids remain fine during the process of SRx, since dispersoid coarsening is suppressed due to the grain boundary break-away process of SRx.[5] The microstructure of sections parallel to the extrusion axis typically consists of well developed elongated, SRxed grains having a grain aspect ratio of 5 (200~500 μm × 1000~2000 μm), as shown in Fig. 1. This type of elongated grain structure is commonly observed after isothermal annealing or zone annealing of ODS superalloys.[4,11]

3.2. On the Creep Behavior of SRxed MA NiAl.

Compressive creep tests were performed on SRxed specimens applying a constant load parallel to the ex-

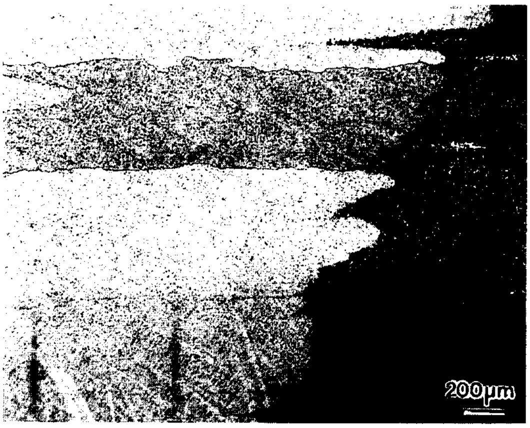

Fig. 1. Optical micrograph of SRx MA NiAl, longitudinal section.

trusion axis ranging from 40 MPa to 180 MPa at 800 ℃, 850 ℃ and 900 ℃ for 20 hours. The normalized creep conditions in this study are $0.56 \leq T/T_M \leq 0.61$ and $4\times10^{-4} \leq \sigma/G \leq 3\times10^{-3}$, assuming no significant differences in shear modulus between as-extruded and SRx conditions. For a representation of general creep trends, creep curves of grain coarsened specimens obtained at 800 ℃, 850 ℃ and 900 ℃ with the stress of 110 MPa are presented in Fig. 2. The creep curves show primary and steady state creep but not tertiary creep in this study, due to the compressive creep mode used, in which most microstructure instabilities such as microcracking or necking are suppressed.[12] Based on the creep curves, the steady state creep rate ($\dot{\varepsilon}_s$) and the total creep strain for SRxed

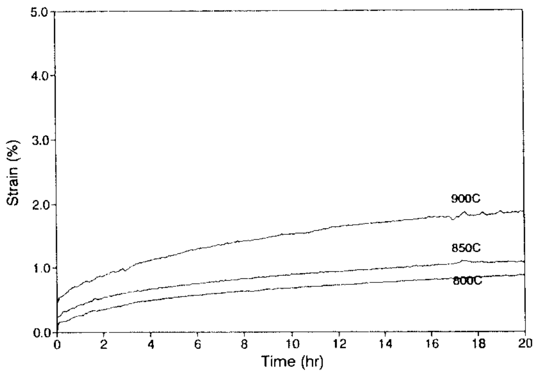

Fig. 2. Creep curves of SRxed MA NiAl, tested at 800 ℃, 850 ℃ and 900 ℃ with the stress of 110 MPa.

Table 1. Steady state creep rates (sec^{-1}) for SRxed MA NiAl

	40 MPa	110 MPa	124.5 MPa@
800 ℃		1.6×10^{-8} (0.55%)*	
850 ℃		6.6×10^{-8} (0.88%)* 4.0×10^{-8} (0.90%)	
900 ℃	6.59×10^{-9} (0.9%)	8.6×10^{-8} (1.43%)* 7.2×10^{-8} (1.7%)	4.57×10^{-7} (31%)

Note: @ stress is a true stress converted from applied stress (180 MPa)
*denotes 2nd set of SRx specimens and the numbers in parenthesis indicate the total creep strain in 20 hrs.

specimens are shown in Table 1.

In order to characterize the creep mechanisms, the activation energy and the creep exponent were calculated based on the creep data[7]:

$$Q_{app} = -R\left(\frac{\partial \ln \dot{\varepsilon}_s}{\partial(1/T)}\right)_{110\,\mathrm{Mpa}} = 177\,\mathrm{kJ/mole} \qquad (9)$$

$$n = \left(\frac{\partial \ln \dot{\varepsilon}_s}{\partial \ln \sigma}\right)_{900℃} = 3.05 \qquad (10)$$

The apparent activation energy for creep in SRx MA NiAl is almost the same as the activation energy (175 kJ/mole) in the as-extruded condition, but the apparent stress exponent for the SRx condition is higher than that (n=2) of as-extruded condition.[2] The activation energy obtained in this study is in the range of the activation energy for the self diffusion of Ni (150~250 kJ/mole) in NiAl,[13] indicating that the creep process is controlled by diffusion regardless of the dominant creep mechanisms.[14,15]

It has been discussed that the creep in SRxed MA NiAl can be considered to be controlled by one of the dislocation creep mechanisms, which are viscous glide controlled or climb controlled dislocation creep.[7] It was reported that the creep for MA NiAl was speculated to be controlled rather by dislocation climb, although the stress exponent seems to fall into the viscous glide controlled mechanism.[7)]

3.3. On the Threshold Stress in SRxed MA NiAl

Creep of ODS materials is characterized by high values of apparent activation energy for creep, and abnormally high values of stress exponent (n~40), and the existence of a threshold stress.[14,15)] It was suggested that the abnormally high values of Q_{app} were attributable to the temperature dependence of the elastic modulus and the abnormally high values of n in dispersion strengthened materials.[15)] The abnormally high n values in DS alloys arise from the presence of a threshold stress which is believed to be originated from the resistance to dislocation motion by dispersoids.[14)] However, SRxed MA NiAl exhibits a normal apparent activation energy for creep and apparent stress exponent.[7)] This is in part due to the relatively insensitive temperature dependence of elastic modulus in NiAl,[17)] and the lower threshold stress value observed in as consolidated MA NiAl.[3)]

Since the creep rate of many ODS alloys is determined by an effective stress (σ-σ_{th}),[8,9,14)] the normalized creep equation with respect to the effective stress is generally expressed by the equation (2). Assuming no significant differences in elastic modulus between SRx and as-extruded condition, an elastic modulus equation, which was experimentally determined for <110> oriented single crystal NiAl,[10)] was applied in this calculation as expressed in equation (6). Based on this equation, the E (900 ℃) and $\partial E/\partial T$ (900 ℃) were found to be 154.1 GPa, -0.0349 GPa/K, respectively. Using n=3.05 and Q_{app}=177 kJ/mole. the true activation energy (Q_c) was estimated to be 169.1 kJ/mole. Thus the Q_{app} measured in SRxed MA NiAl is very similar to the true value because of the relatively insensitive temperature dependence of elastic modulus. A similar result was shown in a previous creep study for as-extruded MA NiAl in which Q_{app} and Q_c are 175 and 174.2 kJ/mole, respectively.[3)]

The threshold stress can be measured by either a load reduction test at constant temperature[18)] or a graphical method[9)] which is used in this study, and the estimated threshold stress by the graphical method shows good correlation with measured threshold stress in the as-extruded condition.[16)]

From the normalized creep equation (2), the threshold stress can be estimated by extrapolating a linear plot of $[\dot{\varepsilon}_s/A_o\exp(-Q_c/RT)]^{1/n_o}$ versus $\sigma/E(T)$ to the zero values of normalized stress. For the plot, n_o is generally present to the normal value of n for dislocation creep (n=4) in the ODS alloys which have abnormally high n value,[14)] but the n_o can be set to 3.05 in this case since the apparent stress exponent is in the normal range of dislocation creep. Q_c is set to 169.1 kJ/mole, A_o is determined from the rearranged creep equation using experimental creep data as expressed in equation (5).

Using the creep data, a linear plot of $[\dot{\varepsilon}_s/A_o\exp(-Q_c/RT)]^{1/n_o}$ versus $\sigma/E(T)$ was made, as shown in Fig. 3, and the zero intercept value was found to be $\sigma_{th}/E_{900℃}$ $=2.2\times10^{-5}$. Hence, putting $E_{900℃}$=154.1 GPa, the estimated threshold stress for SRx MA NiAl at 900 ℃ is found to be 3.4 MPa. Given experimental errors, this result implies that σ_{th} is negligible in the SRxed condition. As a reference, σ_{th} in as-extruded MA NiAl was found to be about 20 MPa.[16)]

As discussed previously, three kinds of models have been put forward to explain the origin and the magnitude of the threshold stress, i.e., Orowan bowing, detachment of dislocation from attracting particles, and localized climb over non-attracting particles.[16)] Among those models, the origin of the threshold stress in as-extruded condition was considered to be the threshold stress for localized climb under equilibrium condition over non-attracting particles.[16)] This model was originally proposed by Rösler and

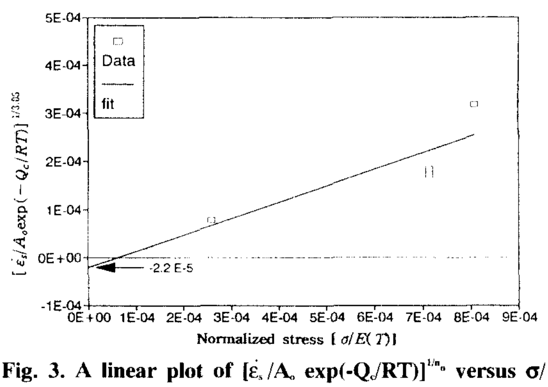

Fig. 3. A linear plot of $[\dot{\varepsilon}_s/A_o\ \exp(-Q_c/RT)]^{1/n_o}$ versus $\sigma/E(T)$ for the threshold stress calculation.

Arzt,[19] and provides the best estimate of the threshold stress in MA NiAl among the currently available models. The threshold stress proposed by this model is given by:

$$\frac{\tau_{th}}{\tau} = \frac{h}{l} \tag{11}$$

where h is the particle height (~particle radius), l is the planar spacing between the particles. The threshold stress can be expected to decrease with particle coarsening assuming no change in volume fraction during the test. Thus, the lower value of threshold stress in the SRxed condition can be attributed to dispersoid coarsening during SRx. It has also been suggested that threshold stress could depend on grain size through the fluctuation of boundary area when the grain boundary sliding accommodation process was considered.[20] In this model, the threshold stress is considered to be the stress necessary to enable deformation to continue and is estimated to be ~0.72γ/d, where d is mean grain size and γ is grain boundary energy.

Using the estimated threshold stress, the stress exponent, which was initially set to 3.05, can be verified from the diffusion compensated, normalized creep equation in the form of:

$$\frac{\dot{\varepsilon}_s}{D} = A_0 \left[\frac{(\sigma - \sigma_{th})}{E(T)} \right]^{n_0}$$

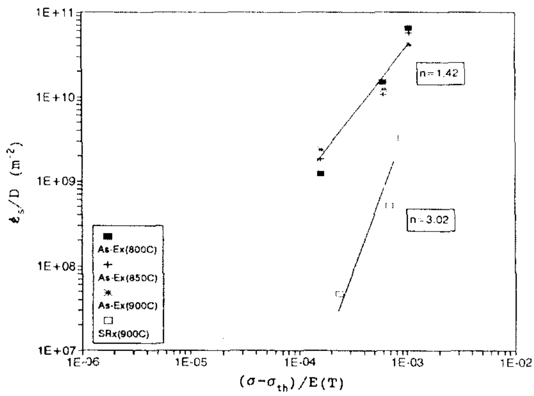

Fig. 4. Diffusion compensated creep rate as a function of modulus compensated effective stress for MA NiAl.

The diffusion compensated, normalized plot is presented in Fig. 4 and n_0 was found to be 3.02 which is almost identical to the apparent stress exponent (n=3.05). This implies that the apparent creep parameters are not very different from the true values due to the low value of threshold stress in SRxed MA NiAl. In the Fig. 4, the plot for as-extruded specimen[16] is also presented for comparison. As can be seen, in comparison to the creep of as-extruded condition, the true stress exponent increases from 1.42 to 3.02, and the effective creep rate was decreased approximately two orders of magnitude by secondary recrystallization. The increase in true stress exponent implies that the contribution of grain boundary sliding accommodation to the dislocation creep in MA NiAl is greater in the fine grained as-extruded condition because the n_0 in the as-extruded condition is close to 1 (diffusional creep), but the contribution becomes smaller due to the reduction of grain boundaries in the SRxed condition leading to high values of n_0.

4. Conclusion

The apparent activation energy and stress exponent for creep in SRxed MA NiAl were found to be 177 kJ/mole and 3.05, respectively. The Q_{app} appears to be in the range of the activation energy for self diffusion and the stress exponent indicates that dislocation creep mechanism is rate controlling.

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The threshold stress is negligible in SRxed MA NiAl. This is attributed to dispersoid coarsening and a decrease in grain boundary area by SRx.

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