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급냉응고된 Al-9.45wt%Fe-4.45wt%Cr합금의 고온 크립거동

Elevated Temperature Creep Behavior of Rapidly Solidified Al-9.45wt%Fe-4.45wt%Cr Alloy

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ABSTRACT

The creep behavior of a rapidly solidified and consolidated Al-9.45wt%Fe-4.45wt%Cr alloy were investigated in the stress range 40 to 115 MPa and temperature range 300(0.53Tm) to 441 ℃(0.66Tm). It is of use to available aerospace and automobile industries for the improved performance of materials used at high temperature. Because Al alloys with improved creep resistance offer the potential for lower weight and reduced costs in aerospace and automobile components (e.g., structural members and engine parts) through the replacement of heavier and more costly materials, the safety in use at high temperature is good. The alloy is characterized by high stress exponents and activation energies for creep, which are greatly dependent on the stress and temperature. Because the creep stress is seen to cause a strongly significant enhancement of coarsening, the coarsening rate of the dispersed particles in all crept specimens is faster than that in isothermally annealed specimens. Dislocations connecting dispersoids are observed more cofrequently in crept specimens with higher stress and lower temperature. The creep strain rates in the power law creep regime were found to be predicted much better by the Sherby and Rösler/Arzt equation with the inclusion of a threshold stress and dislocation detachment mechanism. The dispersoids in this alloy were acting a source of void nucleation that finally leaded to ductile fracture within the grain so called intergranular. Each void was

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initiated, grown and failed at the dispersoids in the aluminium matrix. Grain boundary accommodation of the slip produced, which result in initiation of the void and then final transgranular fracture. Therefore, it was confirmed that these dispersoids played an important role in the fracture mechanism by the formation of $Al_{13}Fe_4$, $Al_{13}Cr_2$ and Al_2O_3.

국 문 요 약

급냉응고 강화된 Al-9.45wt%Fe-4.45wt%Cr 합금의 크립거동을 40~115Mpa 응력범위와, 300~441 ℃(0.53~0.66Tm) 온도 범위에서 조사하였다. 이 계열의 합금은 비행기 및 자동차의 구조용재료 혹은 엔진용 부품에 많이 사용되고 있으며, 재료의 사용이 주로 고온에서 이루어지므로 안전성을 확보하기 위해서는 크립실험이 특히 중요하다. 이 합금의 크립실험 결과 응력지수와 크립 활성화에너지가 높았으며 실험 응력과 온도에 크게 좌우되었다. 크립응력이 조대화에 강하게 영향미치는 것으로 보이기 때문에 모든 크립시편의 분산입자의 조대화율은 등온 소둔시편 보다도 더 빠르게 나타났다. 분산상과 연결된 전위는 고응력, 저온의 크립시편에서 더욱 자주 관찰되었다. Power law creep에서의 크립변형속도는 문턱응력과 전위분리기구를 포함하는 Sherby와 Rösler/Arzt식으로 예견되는 것과 일치함을 발견하였다. 이 합금에서 분산상은 void생성원으로 작용하였으며 소위 입계파괴인 입자내의 연성파괴의 원인이 되었다. 생성된 void는 성장하여 Al기지내의 분산상과 분리되고, 슬립에 의해 결정립계에 집적되어 결국 입계파괴가 일어났다. 그러므로 이들 분산상이 $Al_{13}Fe_4$, $Al_{13}Cr_2$ and Al_2O_3의 형성에 의해 파괴 기구의 중요한 역할을 함이 입증되었다.

1. Introduction

As the demands of aerospace and automobile industries for the improved performance of materials used at high temperature have been increased, a variey of attempts have been made in order to develop an advanced materials for high temperature applications. Aluminium alloys with higher strength and improved creep resistance offer the potential for lower weight and reduced costs in aerospace and automobile components(e.g., structural members and engine parts) thtough the replacement of heavier and more costly materials, such as titanium. Several different compositions and processing techniques are investigated by many researchers and major attention has been focused on pure Al and Al alloys. The investigation of these alloys has been based on hot tensile tests and very little work has done for determining the mechanism of creep of these alloys. Creep resistance is an importance attribute of these alloys. The mechanism that control creep in these alloys must be understood for the design of alloys that resist creep. The creep behavior of pure Al and Al alloys at elevated temperatures is described by a diffusion-controlled processes. These processes are described by a creep equation which shows the apparent stress exponent, $n_{app.}$ of 2~5 when the microstructure changes with the applied stress and apparent activation energy, $Q_{app.}$ associated with that for lattice diffusion in aluminium[1,2]. Most dispersion-strengthened aluminum alloys are interesting in their exceptionally high stress exponents and activation energies for creep usually much higher than those of pure Al and Al alloy[3].

Even though it is well known that dispersoids in Al matrix improve creep resistance, the mechanisms for it are not yet understood clearly. To explain the creep behavior of dispersion-strengthened materials, the power low

creep equation for these materials was introduced[4~6]. It should be noted that the use of this equation means that any influence of dispersoids is limited to the threshold stress as no microstrucual term is included. Sherby et al have proposed a substructure invariant model[1,7]. The basis for such a mechanism is not clear, but it has been argued that only when the substructure is constant, will this model be satisfactor. Recently Rösler and Arzt have proposed a creep model which predicts an exceptional law and it is based on thermal activation detachment of dislocation from dispersoids exerting an attractive force[8]. It is needed to further examine various possibilities which contain the mirostructural details.

A study on the rapidly solidified Al-9.45 wt%Fe-4.45wt%Cr alloy was performed in order to analyse the creep data in the light of both a diffusion-controlled and slip creep mechanism under the presence of a threshold stress and a dislocation detachment mechanism.

2. Experimental procedure

Flat specimens for the creep tests were produced from the as-extruded bar by electrodischarge machining. The longitudinal axis of the specimens was parallel to the extrusion, which is also the long axis of the grains in this alloy. The specimen geometry was with a uniform gage length of 25mm, a gage width of 4 mm, and a thickness of 1mm. To ensure a smooth surface finish, all specimens were hand-polished using 200, 600 and 1,200 grit papers and rinsed in methanol prior to testing. Tensile creep tests for the present study were conducted in a constant stress creep machine designed by Andrade and Chalmer, with test temperatures ranging from 300(573) to 441℃(714K). Specimen elongation was measured with a Schaevitz 1000HR linear variable displacement transducers(below LVDTs) attached to the external machine linkage. Prior to a test, each specimen was heated for about 1 hour until the test temperature was reached and then held for an additional hour to permit the system to reach thermal equilibrium.

Several representative specimens of the Al-9.45wt%Fe-4.45wt%Cr alloy were allowed to creep to the steady-state region, then cooled under load to retain the dislocation structure. The transmission electron microscopy specimens were prepared for examination by cutting thin slices from the deformed gage section to observe a longitudinal section.

3. Results and discussion

Constant stress creep tests of Al-9.45wt% Fe-4.45wt%Cr alloy were performed at various temperatures from 300(0.53Tm) to 441℃ (0.66Tm). Creep data were described in Table 1.

Typical strain vs time plots for tests at 623 K are shown Fig. 1. In all cases, this material exhibited typical curve characteristics of dispersion alloy-type behavior. The results from the tests are shown in Figure as strain rate vs applied stress on double logarithmic axis. The slopes of the curves show very high apparent stress exponents(n_{app}) of above 11 in the equation(1).

$$\dot{\varepsilon}=K\,\sigma^{n_{app}} \quad \cdots\cdots (1)$$

where $\dot{\varepsilon}$ is the strain rate, σ is the applied stress and K is a material constant at a fixed temperature. The flow stress-strain rate data for the alloy are plotted on a log-log scale, with the possibility of the threshold behavior. The bar of material was used for the constant stress tester. As expected for a dispersion-strengthened material, a high apparent stress exponent(n_{app}) is observed. The values

of n_{app}, had already been calculated. We note that the values of n_{app}, varies with the elements and content.

Table 1 Creep data

Temperature (℃)	Stress (MPa)	Steady state creep rate(sec^{-1})×10^{-5}
300	85	0.00141
315	85	0.00188
330	85	0.00246
	100	0.00353
	115	0.00675
340	85	0.00356
	100	0.00578
	115	0.0173
350	85	0.00878
	100	0.01072
	115	0.03031
372	40	0.00497
	45	0.01736
	50	0.0067
	55	0.11889
394	40	0.02314
	45	0.03622
	50	0.06794
	55	0.28761
417	40	0.03472
	45	0.08769
	50	0.17461
	55	0.4664
441	40	0.04903
	45	0.13111
	50	0.32194
	55	1.29722

On the basis of the strain rate-stress data of Fig. 2 at various temperatures, it is possible to determine the activation energy Q for creep such that

$$Q_{app} = -R\frac{\ln\left(\frac{\dot{\varepsilon}}{RT}\right)}{\frac{1}{T_1}-\frac{1}{T_2}} = R\frac{d\ln\dot{\varepsilon}}{d\left(\frac{1}{T}\right)} \quad \cdots\cdots (2)$$

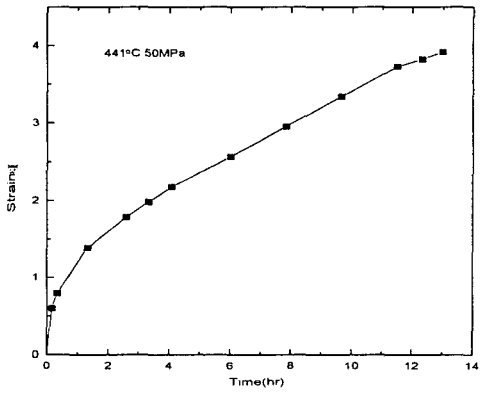

Fig. 1 Typical creep curve of Al-9.45wt%Fe-4.45wt%Cr alloy

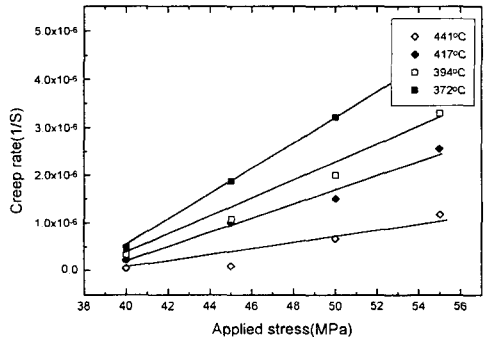

Fig. 2 Steady state creep rate as a fuction of applied stress for Al-9.45wt% Fe-4.45wt%Cr alloy at various temperatures from 372 to 441℃

where R is the gas constant and T is the absolute temperature. The apparent activation energy is calculated by this equation. Generally, Q_{app} increases with decreasing stress and n_{app} decreases with increasing temperature. The apparent activation energy for creep of this material is as high as 310 kJ mol^{-1} at 441℃. This value is significantly higher than the activation energy for lattice self diffusion in aluminum (Q_L), which is equal to 142 kJ mol^{-1} [9]. The apparent stress exponent is also very high; the minimum n_{app} value, measured at the highest testing temperature, 441℃, is n_{app}=11. In consideration of threshold stress,

true stress exponent may be about 5 at 350℃.

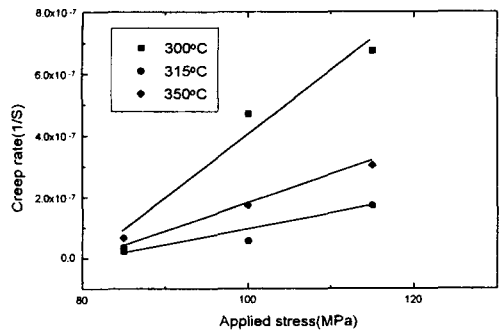

Fig. 3 Steady state creep rate as a fuction of applied stress for Al-9.45wt%Fe-4.45wt%Cr alloy at various temperatures from 300 to 350℃

The microstructure of the Al-9.45Fe-4.45Cr extrudate consists of fine grains about 0.5 μm in size stabilized by fine intermetallic particles of Al_6Fe, $Al_{13}Fe_4$ and $Al_{13}Cr_2$ about 20nm in diameter. The dispersoids possess a round, needle and plate morphology and are thermally stable. TEM observation of the samples tested showed some coarsening of dispersoids or changed in particle spacing and grain size after deformation. Special attention was paid to the interface between the dispersoid particles and matrix. A TEM bright-field micrograph is given in Fig. 4, which corresponds to a sample tested at 350℃ and 85MPa. This microstructure consists of fine grains containing small slightly elongated subgrains and loosely tangled dislocation, as apparent in Fig. 4. Despite the high-creep temperature, the specimen still possesses the dislocation in the form of dislocation tangles, that remain visible in grain interiors. The grain size of the specimen tested at 350℃ and 85MPa is slighter larger than that of a specimen test at a higher stress level, at the same temperature, mostly likely due to the difference in applied stress. The microstructure of this dispersion strengthened alloy shows the frequent particle /dislocation interactions that occur during creep. Examples of dislocations attached to Al_2O_3 and dispersoids are specifically identified The specimen tested at 300℃(573 K) and 115 MPa appears to develop essentially equiaxed grains. However some grains appear slightly elongated. This is in contrast to the predominace of elongated grain for the specimens tested at high temperature and low stress. Dislocation density remains high in the interiors of grains. The grain size is only marginally sensitive to temperature or applied stress.

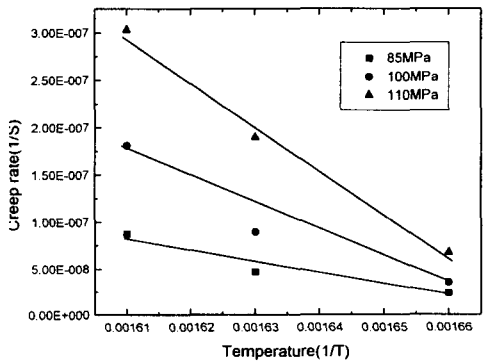

Fig. 4 Apparent activation energy for creep of Al-9.45 wt%-4.45wt%Cr, Q_{app} was calculated to be 268 kJ/mol, while the activation energy for selfdiffusion of self diffusion of Al was only 142kJ/mole

This slight difference in size suggests that grains do not form as a natural response to the applied creep stress, in contrast to the behavior of pure polycrystalline Al, which exhibits an inverse dependence of grain size on stress. Unlike Al_2O_3 and dispersoid free material, in which the dislocation-dislocation interaction distance and grain size are discovered by the stress, the creep behavior and the substructure of the dispersion-strengthened Al are dominated by particle dislocation interactions. These interactions represent the direct influence of particles on the creep be-

havior. This interpretation is supported by clear evidence of particle/dislocation interactions observed in all specimens shown in Fig. 4. By analogy to the theoretical treatment by Holt[10,11], the indirect role of the particles is to increase the resistance to glide, which produces a smaller gain size. In Al-9.45Fe-4.45Cr alloy, the particle-dislocation interactions most likely prevent the extent of dislocation motion needed to develop a well-defined subgain structure. This limitation gives rise to a subgrain structure which is not completely developed, but rather consists of diffuse, poorly defined boundaries. Further, most grains are not fully equiaxed, especially for the high temperature-low stress cases, adding evidence that the microstrucure is only approaching steady-state conditions. Under these conditions, characterization of grain size is difficult. As seen in most of the figures from the present investigation, particles are distributed both within gains and at grain boundaries, suggesting that the formation of a characteristic steady-state creep substructure is inhibited by the presence of particles.

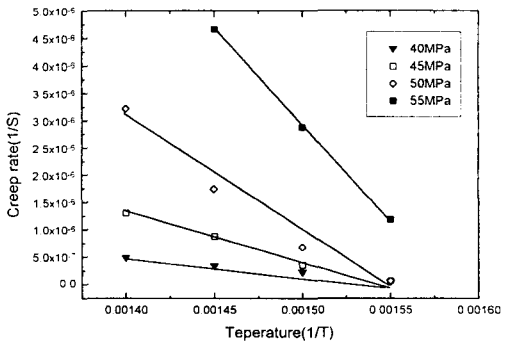

Fig. 5 Apparent activation energy for creep of Al-9.45 wt%-4.45wt%Cr, Qapp was calculated to be 310 kJ/mol, while the activation energy for self diffusion of Al was only 142kJ/mole

Based on the microstructural observations presented above the sentence, the Rösler-Arzt model of dislocation detachment controlled creep appears to be applicable to the results for Al-9.45wt%Fe-4.45wt%Cr alloy. In order to analyze the data according to this theory, it is necessary to experimentally determine values for the apparent stress exponent, n_{app} and the corresponding apparent activation energy for creep, Q_{app}. The change in n_{app} from the lower temperatures, 300 to 350℃, to the higher temperatures, 372 to 441℃. Suggests that Q_{app} should be determined separately for each region. Initially, tests were only performed at 372(645) to 441℃. To accurately determine Q_{app}, several additional tests were performed at 372 and 441℃ and at several stresses to complement the data at 300 and 350℃. The apparent activation energy was calculated by equation(2) in which the temperature-dependent modules data of W. Koester and Z. Metallkd were used. In order to avoid errors in analysis associated with possible nonlinearity in the ln $\dot{\varepsilon}$ vs ln σ plots at low strain rates, only tests at strain rates above $10^{-5}s^{-1}$ were used in the calculation of the apparent activation energy, as well as in the remainder of the Rösler/Arzt theory calculations. Further, only the data from the present study were used in these calculations. The most notable aspect of these calculations is the close agreement in Q., between the low temperature (268.7kJ/mole) and high temperature (310.9kJ/mole). Generally, an increase in the activation energy for creep, Q_{app} of pure, polycrystalline Al occurs as the temperature is increased above approximately 350℃(423K). The choice of an appropriate activation energy for vacancy diffusion, Q_v, with which to compare the values of Q_{app}, is not immediately obvious. With the heavily dislocated structure and fine grain size of this material, the options for appropriate activation energies are

expanded to include lattice self-diffusion, dislocation core diffusion, and grain boundary diffusion. The measured value of Q_{app}, is greater than all of the activation energies for the self diffusion mechanisms. This is a common characteristic observed in the creep of dispersion strengthened metals. As previously noted, the high dislocation density inherited from processing is not easily recovered at the test temperature used in the present investigation. Thus, the possibility of diffusion of vacancies to and from the particle/dislocation interface via dislocation cores is easily achieved. As a consequence, grain boundary diffusion may play an important role in the process of supplying vacancies to the point of detachment within subgrain interiors. The activation .energy for this mechanism would be equal to that for grain boundary diffusion, which is not very different in value from the activation energy for dislocation core diffusion. Thus, it is not possible to unambiguously rule out grain boundary diffusion as the rate-controlling transport step. However, selfdiffusion through the aluminium lattice should be less effective in delivering vacancies to the detachment site than either grain boundary or core diffusion. Based on these considerations. it is not obvious which mode of vacancy transport controls the thermally activated detachment process. Accordingly, lattice self-diffusion, grain boundary diffusion and dislocation core diffusion are all analyzed with respect to the application of the model of thermally activated detachment. The ratio of the applied stress to the detachment stress for the Rösler-Arzt model was calculated using the expression

$$\frac{\sigma}{\sigma_d} = \left(\frac{3(Q_{app} - Q_v)}{2RT\, n_{app}\left(1 - \frac{dG}{dT}\frac{T}{G}\right)} + 1 \right)^{-1} \quad \cdots\cdots (3)$$

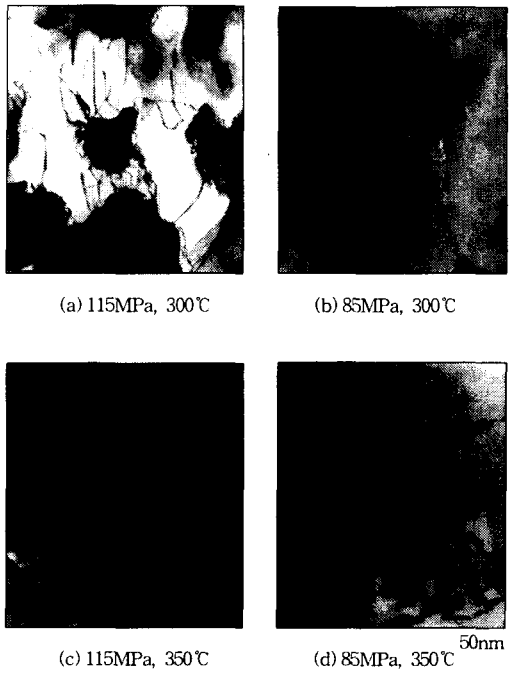

Fig. 6 TEM of the crept specimens

The interaction parameter, k, was then calculated according to the relationships

$$k_A = 1 - \left(\frac{2kT}{3G} b^2 r \frac{n_{app}}{[1-(\sigma/\sigma_d)]^{1/2}(\sigma/\sigma_d)} \right)^{2/3} \quad \cdots\cdots (4)$$

based on the average particle radius of 5nm. The value of k does notice significantly with the choice of vacancy transport mechanism. Also, the values of k at the different temperatures are consistent, with an overall average value of approximately 0.9. As previously stated, the limit for detachment control has been theoretically determined to be k=0.94 while k=0.9 has been considered a critical value for practical applications. Thus, dislocations interact with the internal Al_2O_3 and dispersoid in this material, although the interaction is only moderately strong. This result is consistent with the observation that the particle/matrix interface is partially coherent in

$Al-Al_2O_3$ alloys. The values for k at each temperature were used with the equation

$$\sigma_d = \sigma_{or}\sqrt{1-k_A^2} \quad \cdots\cdots (5)$$

to calculate the detachment stress, σ_d for the creep conditions studied in the present investigation The slight drop in detachment stress between 300 and 441℃ can be attributed to the temperature dependence of the elastic modulus, which also accounts for the decrease of the Orowan stress, σ_{or}, with increasing temperature. We noted that the subgrain boundaries varied from loosely tangled dislocations to well-defined arrays. In addition, a relatively low density of dislocations within subgrain interiors was observed, with evidence of attractive interactions between particles and dislocations.

4. Conclusions

The creep behavior of an available dispersion strengthened aluminium alloy has been examined in the temperature range of 300 (0.53Tm) to 441℃(0.66Tm). The results of this observation reveal the following:

1) High dislocation densities and dislocation tangles are observed within grain interiors. Only a marginal influence of the applied stress level on grain size was observed.
2) Particle/dislocation interactions were frequently observed to be the dominant factor in controlling the creep behavior.
3) Apparent activation energies of 268.7 to 310.9kJ/mole and high apparent stress exponents of 11 to 17 were measured, in agreement with data for other dispersion strengthened metals such as Al-Ti and $Cu-Al_2O_3$.
4) The creep data can be described by the model based on an attractive interaction between dislocations and dispersoids developed by Rösler and Arzt.

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