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Some aspects of anelastic and microplastic creep of pure Al and two Al-alloys

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Abstract - Anelastic creep of pure Al, commercial Al-Cu and a binary Al-Cu alloy has been measured at room temperature by means of a high resolution laser interferometer. The irreversible component of the deformation was also quantified from measurements of the anelastic creep recovery. The dependence of the deformation-time curves on thermal treatment and cold work is analyzed. The mechanisms responsible for the room temperature anelastic creep are discussed. Materials loaded below their elastic limit can present either a pure anelastic behavior (commercial Al-Cu) or additional viscoelastic creep (pure Al, high purity Al-Cu). For commercial Al-Cu, the presence of an irreversible deformation appears to be mainly related to the state of the surface. A viscoelastic after effect has been measured for this alloy after a Cu-electroplating treatment. As a typical result for room temperature creep, the irreversible deformation depends logarithmically on load time.

1. INTRODUCTION

The future nanometer-dimension technology requires highly stable and drift-free materials. Therefore, we have analyzed the room temperature anelastic creep of pure Al and Al-Cu alloys by means of a high resolution laser interferometer [1]. This enables a resolution of flexural displacement of about 0.1 nm, which corresponds to a strain resolution of some 10^{-10} for our sample geometry. The aim of the present work is to establish the microstructural causes of the anelastic and microplastic creep of the above mentioned alloys, and to study the influence of the alloy composition, the thermal treatments, the state of the surface on room temperature creep.

The anelastic behavior of Al and Al-alloys, mainly measured by internal friction, has been the object of several investigations. The internal friction curves, measured as a function of frequency or temperature, provide an easy way for separating the different dissipation mechanisms. On the other hand, direct creep measurements supply valuable information on the long term stability of loaded materials (like spring or bearing elements) and allow to distinguish between reversible and irreversible contributions to the deformation.

2. EXPERIMENTAL RESULTS: ANELASTIC CREEP CURVES AND ASSOCIATED MICROSTRUCTURES

2.1. Reversible and irreversible creep of commercial Al-Cu

A typical load-unload cycle of a Cu-electroplated commercial Al-Cu sample is presented in Fig. 1. The applied load corresponds to 1/10 of the elastic limit of the alloy. The measurement of the elastic after-effect allows to distinguish between pure anelastic and viscoelastic beha-

rior and to establish the time-dependence of irreversible deformation. As typical for room temperature visco-elastic creep, a logarithmic dependence of the irreversible component from load time has been observed, while the anelastic creep nearly follows a power law. The irreversible displacement (η_{irr}) evaluated as a function of time t by measuring the non-recovered displacement at the end of every cycle, is shown in Fig. 2. A similar logarithmic dependence of irreversible deformation on time has been shown to be valid even for pure Al and a commercial Al-Zn alloy [2].

Fig. 1: Load-unload cycle of a Cu-electroplated sample of commercial Al-Cu. A part of the displacement cumulated during loading remains unreleased.

Fig. 2: Irreversible displacement plotted as a function of the total time under load for Cu-electroplated commercial Al-Cu

Fig. 3: Anelastic creep curves (loading) for pure Al-3.8%Cu, measured for different thermal treatments at 203 °C.

2.2. Anelastic creep of Al-3.8%Cu

Fig. 3 presents displacement-time curves for the loading of artificially aged Al-3.8%Cu samples. The aging temperature was 203°C. Samples aged for times between 1/2h and 136h were measured. The anelastic behavior of the alloy depends on aging time. The time dependence of the displacement follows roughly a simple power law with exponent between 0.35 and 0.5, depending on the aging state. For the intermediate aging time of 9h, TEM investigations have shown the presence of GPII zones and θ' precipitates (Fig. 4) and a network of dislocations preferentially pinned on the precipitates (Fig. 5).

Fig. 4: a) TEM micrograph (Bright Field) showing GPII and θ' precipitates in artificially aged (190 °C, 9h) Al-3.8%Cu; b) Dark Field (DF) of the θ' precipitate in Fig. 4a. ZA= $\langle 100 \rangle$, DF from the $\langle 011 \rangle$ spot.

Fig. 5: Dislocation structure in artificially aged (190 °C, 9h) Al-3.8%Cu. ZA= $\langle 110 \rangle$, $s=0$.

2.3. Pure Al (Al 1199)

We have studied the effect of an annealing and of cold work on the anelastic creep of pure Al (4N purity, grain size 5mm). In Fig. 6a we show the effect of a 200 °C annealing applied to an as-cast sample, while in Fig. 6b the same effect is shown for a sample which was cold worked before the annealing treatments. The difference in the displacement values between Fig. 6a and 6b is due to different stress levels. The stress level applied to obtain the results of Fig. 6b gave rise to a small viscoelastic deformation during loading. We therefore plot the unloading curves, which are of pure anelastic nature as the curves shown in Fig. 6a. From the results presented it appears that anelastic creep is reduced by annealing, and that longer annealing times correspond to lower anelastic relaxation.

Three microstructures associated to three different partially annealed states are shown in Fig. 7. Different dislocation structures and densities correspond to different anelastic behaviors: as cast specimens present high density of free dislocations (Fig. 7a); with increasing aging time, the dislocations first tend to group in linear walls (Fig. 7b), and finally are mostly annealed. Only some long dislocation segments are observed after 6h annealing (Fig. 7c).

Fig. 6: a) Load curves for as cast pure Al 1199 measured at 22 and 42 °C; b) Unloading curves measured at 22 °C for the same alloy, but for a sample of different geometry, showing viscoelastic behavior during loading.

The three mentioned dislocation structures correspond to a progressive reduction of anelastic creep. On the other hand, cold work enhances anelastic creep, while multiple cycling at a stress not exceeding the elastic limit reduces it. An example of this reduction is shown by the two lowermost curves of Fig. 6b.

Fig. 7: Typical dislocation structures observed in Al 1199: a) as cast; b) 4h annealed at 200 °C; c) 6h annealed at 200 °C. $ZA \langle 110 \rangle$, $s > 0$.

3. ANALYTIC INTERPRETATION OF THE ANELASTIC CREEP

The impossibility of describing any of the measured displacement-time curves in terms of a simple exponential (Voigt model) suggests that several relaxations are acting. Therefore, a spectrum (1) with multiple characteristic times, discrete or continuous, is required for interpreting the curves. A continuous spectrum can be obtained through an inverse-Laplace transformation of the measured curve [3] and a discrete through a tile addition method [4]. Both the spectral analyses are unfortunately intrinsically unstable [5]. The use of an approximate method is then necessary. The second order Schwarzl method [6] has been applied to obtain the approximate spectra. In particular we show in Fig. 8 the spectra obtained from three of the curves reported in Fig. 3 for Al-3.8%Cu. In correspondence with the known characteristic times for the θ' controlled relaxation and the Zener relaxation, some peaks appear in the spectra. Their relative heights depend mainly on aging time: with increasing aging time, the θ' peaks are rising and the Zener peak disappears. The rise of the average level of the spectra with increasing aging time corresponds to the increase of the background in internal friction and is interpreted as due to θ precipitation [7]. Fig. 9a and 9b show the spectra for different curves of pure Al. The good agreement of two curves relative to slightly different annealed Al samples (10 min and 30 min at 200 °C, respectively), which present several peaks of the same heights and located at similar relaxation times, confirms the stability of the applied approximate method of spectral decomposition.

Fig. 8: Second order Schwarzl spectra of Al-3.8%Cu, evaluated from three of the loading curves of Fig. 3.

(1) The spectral decomposition of a curve $\eta(t)$ is:

$$\eta(t) = \int_0^{+\infty} G(\ln\tau) \cdot (1 - \exp(-t/\tau)) d\ln\tau$$

where $G(\ln\tau)$ can be a continuous or discrete spectrum

Fig. 9: Second order Schwarzl spectra of Al 1199, evaluated from: a) some loading curves of Fig. 6a; b) some unloading curves of Fig. 6b.

4. COMPARISON WITH INTERNAL FRICTION MEASUREMENTS

4.1 Al-3.8%Cu

Complementary informations about relaxation can be obtained from measurements of internal friction (Q^{-1}). Fig. 10 shows the logarithmic decrement-temperature curve measured by Berry and Nowick [7] for an Al-Cu alloy of about the same composition and purity of our Al-3.8%Cu. Fig 11 presents the corresponding anelastic creep measured at 22 °C and 42 °C, as well as the static response obtained by transforming the results of [7]. In order to accomplish the transformation, the Zener peak was assumed as a simple relaxation peak, while the θ' peak was approximated by a set of five Debye peaks. The characteristic times at room temperature were obtained by the given activation energies. The experimental static behavior of the alloy at room temperature appears to be perfectly reproduced and even the anelastic creep behavior at $T=42.6$ °C can be predicted (Fig. 11).

Fig. 10: Logarithmic decrement plotted vs. temperature for Al-4%Cu [7]. The results are resolved into a Zener peak and a θ' -peak.

Fig. 11: Anelastic creep of 190 °C-9h aged Al-3.8%Cu. The experimental curves are reported together with the curves obtained from the conversion of the results of Fig. 9. The model is also able to reproduce the behavior at 42.6 °C.

Fig. 12: a) Anelastic creep and b) internal friction-frequency curve for pure Al. The experimental values of internal friction evaluated in a forced pendulum are plotted together with the response obtained by transforming curve a)

4.2. Pure Al

Fig. 12a shows the anelastic creep of pure Al, while in Fig. 12b we plot the room temperature internal friction curve measured as a function of the frequency ν for the same material at room temperature in a forced torsional pendulum. From the measured creep of Al (Fig. 12a), we have derived and reported in Fig. 12b the corresponding Q^{-1} - ν curve. This conversion consists essentially of the Fourier transform of the time derivative of the static creep response. For mathematical details on the procedure of conversion of anelastic response functions, see [3]. The experimental Q^{-1} - ν curve superposes well to the curve deduced from the static measurement. The accord between the results of the two types of experiments, verified as well for Al-Cu and for pure Al, confirms the experimental possibility of interpreting the load curves in terms of the corresponding dynamic internal friction curves.

5. INTERPRETATION OF THE ROOM TEMPERATURE ANELASTIC CREEP

Despite the apparent similarities between the anelastic or the viscoelastic creep curves of different kinds of materials, and in particular of different Al-alloys, the interpretation of the anelastic behavior of metals requires a detailed discussion of the possible mechanisms active in the different alloys.

5.1. Al-3.8%Cu

From the results of: a) direct analysis of creep curves; b) second order Schwarzl analysis of creep data; c) measurement of internal friction and deconvolution of the well known θ' and Zener relaxation peaks it can be confirmed that:

- 1) the θ' relaxation mechanism is mainly active at shorter times ($0.1 \text{ min} < \tau < 2000 \text{ min}$). θ' relaxation is enhanced by longer aging at 203 °C
- 2) the Zener relaxation is active at longer relaxation times ($t > 2000 \text{ min}$). The heights of Zener peaks decrease with longer aging times. Deconvolution of internal friction data suggests that the Zener peak is a pure Debye peak which at room temperature should shift up to $\tau = 15000 \text{ min}$. Spectral analysis (Fig. 8) allows to find the beginning of the Zener phenomenon already at shorter times.

5.2. Pure Al 1199

The strong decrease in internal friction with annealing at 200 °C, as well as the increase due to cold work suggest that the room temperature anelastic creep of pure Al is mainly related to dislocation phenomena. TEM micrographs have shown that the as-cast state corresponds to a high density of mutually pinned and of free dislocations, while successive annealings allow the dislocation density to decrease and the remaining

dislocations to arrange themselves in linear walls and then into long segments of isolated dislocations. This evolution of dislocation structure corresponds to the observed decrease of anelastic relaxation. The spectral analysis reveals that during annealing the heights of the peaks decrease monotonically with increasing aging time, while the position of the peaks and their number stay almost constant. The presence of several relaxation peaks suggests that multiple relaxations are acting: different dislocation lengths may correspond to different times necessary to relax, with longer dislocations requiring longer times. That explains why, in passing from the cold worked state to the annealed state the short time peaks, mainly present in the cold worked state, are definitively reduced by annealing (short dislocation segments disappear), while the peaks at long relaxation times are only reduced (long dislocation segment, despite the general decrease in dislocation density, still survive). Without entering into a too detailed interpretation, which would be premature on the basis of the data of an approximate spectral analysis, the additional result that cold worked samples are subject to high damping (Köster effect [8]) can be interpreted at a first approximation by the same type of dislocation damping which is roughly proportional to the dislocation density. Annealing, on the other hand, reduces the dislocation density and, therefore, the damping capacity. Finally, we remember that the anelastic behavior of pure Al could not be entirely interpreted, in the room temperature and low frequency range, in terms of a vibrating string model [9]. Mason and Wehr suggested, for low frequency range, a model in terms of motion of dislocation kinks across Peierls type barriers [10].

6. CONCLUSIONS

Despite the similar aspect of room temperature anelastic or viscoelastic creep curves of the tested materials, their anelastic behavior depends on phenomena which must be individually analyzed and separated. The combined use of different investigation methods (high resolution anelastic creep measurements, internal friction, TEM observations) and analytical procedures (spectral analysis) has permitted to identify the probable mechanisms which are responsible for anelastic creep at room temperature. The creep of pure Al-3.8%Cu has been interpreted as due to known θ' and Zener relaxation mechanisms. It has been shown that the state of the surface has an important influence on anelastic creep of industrial Al-Cu, and that a surface treatment can induce important irreversibilities. Finally, the influence of the dislocation structure on anelastic creep of pure Al has been pointed out.

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