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Analysis of the anelastic creep of Al and two Al–Cu alloys

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Abstract

Anelastic creep of pure Al, Al–Cu and Al–Cu–Mg was measured at room temperature by means of a high resolution laser heterodyne interferometer. The anelastic deformation was studied as a function of time for several thermomechanical treatments. The obtained creep functions roughly follow over times of up to several days a power law with exponents between 0.3 and 0.5. The relaxation strengths depend strongly on the material and the thermomechanical treatment. An approximate spectral analysis of the creep curves provides valuable information on the form of the spectrum. The static anelastic behaviour and the dynamic response of the same materials are quantitatively compared, and a good agreement between the two types of result was obtained. In particular, for artificially aged Al–Cu, the static behaviour of the alloy could be predicted from the internal friction measured as a function of temperature. The nearly perfect agreement between the two curves indicates that the static room temperature response is governed by the same relaxation mechanisms (Zener and a θ' relaxation) as are responsible for internal friction at higher temperatures. Microstructural transmission electron microscopy observations confirm this interpretation.

1. Introduction

Drift-free materials are required in several fields of current technology. Improving their stability on a nanometric scale is one of the goals of future nanometre-dimension technology. In order to study the stability of spring materials under constant load, we have analysed the room temperature anelastic creep of several metals and alloys by means of a heterodyne laser interferometer [1]. This allows a resolution of flexural displacement of about 0.1 nm, which corresponds to a strain resolution of some 10^{-10} for our sample geometry. Besides the traditional spring materials such as Cu–Be and ferrous alloys, Al alloys also have an increasing importance in the production of spring elements. In the present work we focus attention on pure Al and Al–Cu alloys. The influence of the different microstructures associated with different alloy compositions and thermomechanical treatments is studied.

The results of anelastic creep measurements are compared with the internal friction curves. The anelastic behaviour of pure Al and Al–Cu alloys, 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 to separate the different dissipation mechanisms. Moreover, direct creep measurements supply valuable information on the long-term stability of loaded materials and allow us to distinguish between reversible and irreversible contributions to the deformation.

2. Experimental results

A typical load–unload cycle of a binary Al–3.8 wt.%Cu sample is presented in Fig. 1. The alloy was cast in the laboratory from 99.999% pure Al and Cu. The applied load corresponds to 1/10 of the elastic limit of the alloy. The alloy shows viscoelastic behaviour. Al–Cu–Mg (Al 2024) has pure anelastic behaviour, while pure Al, binary Al–Cu, commercial Al–Zn and

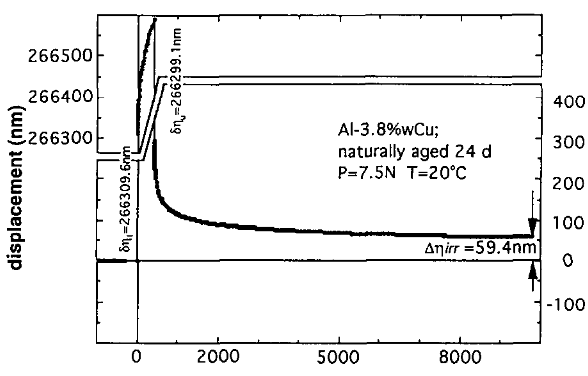

Fig. 1. Loading–unloading cycle of Al–Cu. The instantaneous elastic displacement during loading $\delta\eta_l$ and unloading $\delta\eta_u$ as well as the irreversible deformation $\Delta\eta_{irr}$ are precisely measured.

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sand-blasted or Cu-electroplated Al–Cu–Mg present, under the same load conditions, a non-recovered deformation at the end of every cycle. For 99.99% pure Al and commercial Al–Zn, as is typical for room temperature viscoelastic creep, a logarithmic time dependence of this irreversible component is observed, while the anelastic creep nearly follows a power law. A similar logarithmic dependence of irreversible deformation on time is observed for Cu-electroplated Al–Cu–Mg [2].

Figure 2 presents displacement–time curves for the loading of naturally aged Al–3.8wt.%Cu samples. Measurements were made on specimens aged for between 5 h and 24 days. Between cycles the free sample was allowed to recover. The time dependence of the displacement roughly follows a simple power law with an exponent of about 0.5. With increasing aging time, a decrease in the relaxation strengths is observed. Figure 3 shows the effect of artificial aging at 203 °C on the anelastic creep of the same alloy. It appears from Fig. 3 that with increasing aging time the anelastic strengths at short times (within a few minutes) increase and decrease at long times, with the exception of the uppermost curve for 0.5 h aging. The curve for 1 h aging, which is the lowest at the short measurement times, crosses the other curves at the long times. As for the naturally aged sample, the creep behaviour could roughly be described by a power law with exponent between 0.35 and 0.5, despite the specific characteristics of the different curves.

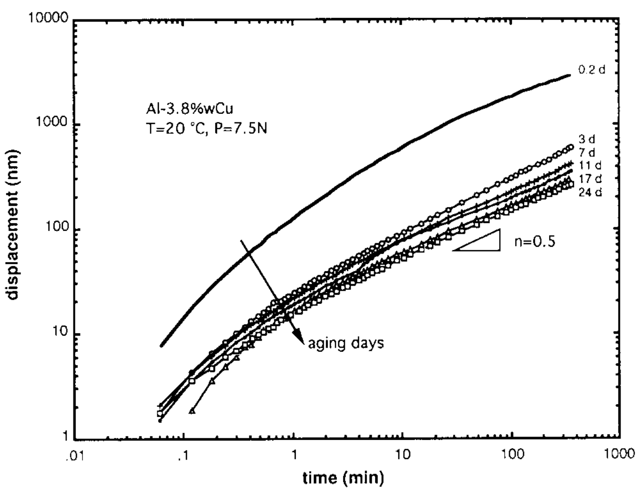

Fig. 2. Loading curves of Al–Cu, measured during natural aging.

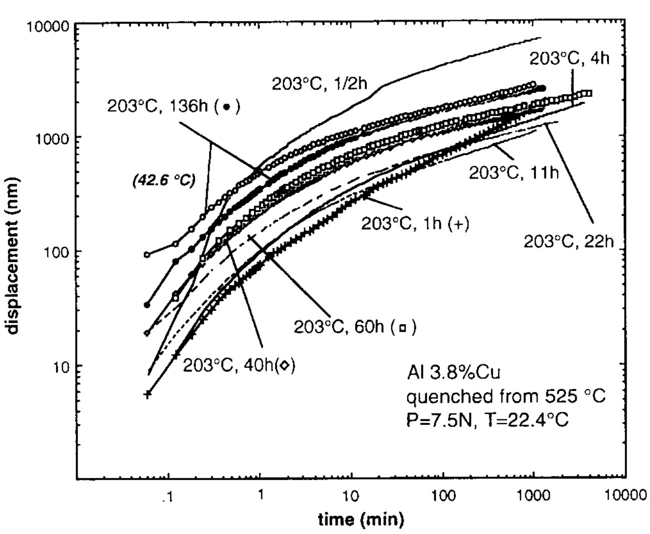

Fig. 3. Loading curves of Al–Cu aged at 203 °C.

The effect of a thermal treatment at 120 °C for commercial Al–Cu–Mg is shown in Fig. 4. As in the case of binary Al–Cu, the artificial treatment has an influence on the anelastic creep behaviour. Under the same load and temperature conditions, Al–Cu–Mg has lower relaxation strengths than binary Al–Cu. All the curves closely follow a power law with exponent between 0.3 and 0.4. As we have shown in a previous paper [3], cold work applied before aging would have slightly increased the exponent of the power law describing the creep behaviour of the alloy.

3. Spectral analysis of anelastic creep curves

None of the measured creep curves can be described in terms of a simple exponential. A simple exponential behaviour would allow one to describe the anelastic creep of a material in terms of a single Voigt unit [4]. The generalization in the discretum or in the continuum of the Voigt model, taking into account a sequence of Voigt units, permits a description of the displacement response $\eta(t)$ for constant load in terms of an integral sum of exponential functions

$$\eta(t)=\int_{0}^{+\infty} G(\ln\tau)[1-\exp(-t/\tau)]\,\mathrm{d}(\ln\tau) \tag{1}$$

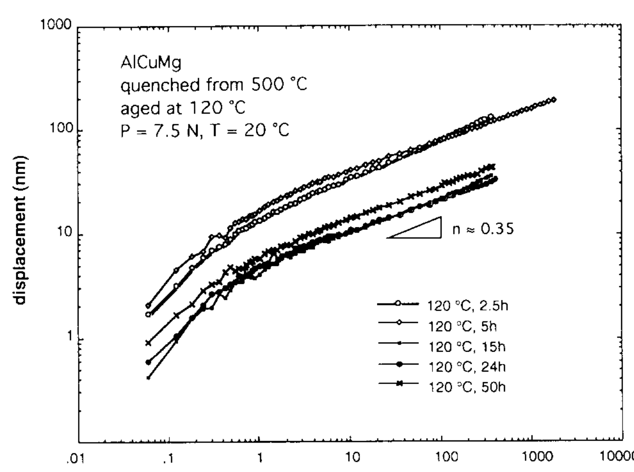

Fig. 4. Loading curves of Al–Cu–Mg aged at 120 °C.

where $G(\ln \tau)$ can be a continuous or a discrete spectrum. The analytical difficulties in finding the spectrum $G(\ln \tau)$ were described elsewhere [3]. They essentially arise from the instability of inverting a Laplace transform [5]. We then considered two approximate methods of spectral decomposition. The application of the method described by Bellman *et al.* [6], which consists of a numerical inversion of the Laplace transform, is presently being studied. Here we apply the method proposed by Schwarlz and Staverman [7] to the experimental creep data of Al–Cu alloys. We used the second-order approximation, which gives the spectral function $G(\ln \tau)$ as

$$G(\ln \tau) \approx \left[\frac{d}{d(\ln t)} - \frac{d^2}{d(\ln t)^2}\right]\eta(t)\Big|_{\tau=(1/2)t} \qquad (2)$$

In particular, we show in Fig. 5 the second-order Schwarzl spectra obtained from three of the curves reported in Fig. 2 for naturally aged Al–3.8%Cu. At long times in the range where Zener relaxation is active [8] we obtain a peak (labelled III) which decreases with aging time. Similarly, the two other peaks I and II decrease on aging. We do not know to what extent the significance of the position and heights of the single peaks can be interpreted. The stability of the peak positions when changing the aging time is an argument in favour of the reliability of the approximate method. In Fig. 6 we show the spectra calculated for artificially aged Al–Cu, derived from Fig. 3. Again in the range of Zener relaxation the relaxation strength decreases with aging time, whereas in the time range characteristic of θ' relaxation the relaxation strength increases.

The results of the spectral decomposition of the anelastic creep curves measured for artificially aged Al–Cu–Mg (Fig. 4) are reported in Fig. 7. Groups of similarly positioned peaks (labelled a, b, c and d) can be found on the curves relative to different aging times. The positions of the peaks of other spectral curves relative to intermediate aging times (not shown in the figure) appear at the same positions. The relaxation strength at long times (d peaks) decreases during the first 15 h of aging. Since, during the first hours of aging, most of the Cu precipitates, this might be the lower end of a Zener relaxation. With further aging, other precipitates probably increase the general level of the curve. As for the d peaks, the heights of the other peaks do not depend monotonically on aging time.

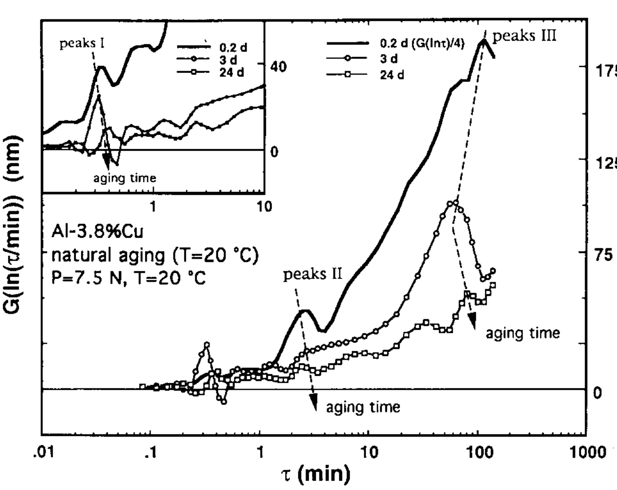

Fig. 5. Spectra associated with the anelastic creep curves of naturally aged Al–Cu (Fig. 2). Note that the curve for 0.2 days is in one case scaled by 4.

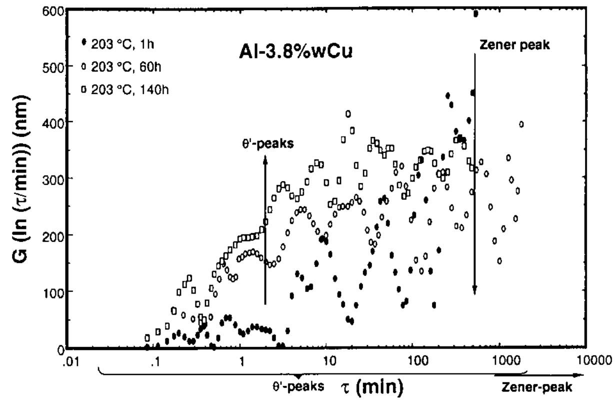

Fig. 6. Spectra of artificially aged Al–Cu, evaluated from three of the loading curves of Fig. 3.

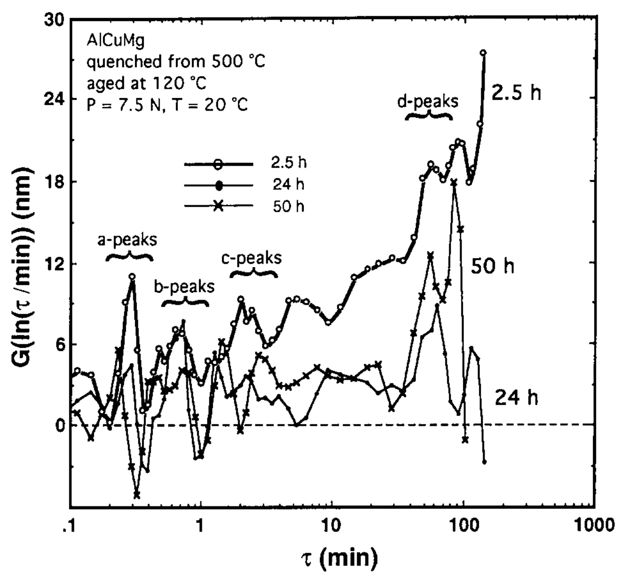

Fig. 7. Spectra of artificially aged Al–Cu–Mg.

4. Results of microstructural observations

For binary Al–Cu, at the intermediate aging time of 9 h at 190 °C, transmission electron microscopy (TEM) investigations showed the presence of θ' precipitates.

For the same aging conditions, Fig. 8(a) shows a structure of dislocations preferentially pinned on the precipitates. Al–Cu–Mg (aged at 120 °C for 24 h) presents, corresponding to a state of minimum relaxation strengths, a network of short dislocation segments and an extremely fine precipitation (Fig. 8(b)). The precipitates are probably able effectively to pin the dislocation segments. An important difference in the mean relaxation strengths for the two types of alloy may be attributed to their different dislocation structure. The slight difference in their average grain size (24.4 μm for Al–Cu instead of 8 μm for Al–Cu–Mg) can hardly justify the different creep behaviour: grain boundary effects are strongly reduced in alloys with grain boundary precipitation [9], and also for pure Al they should intervene at times beyond our range of observation.

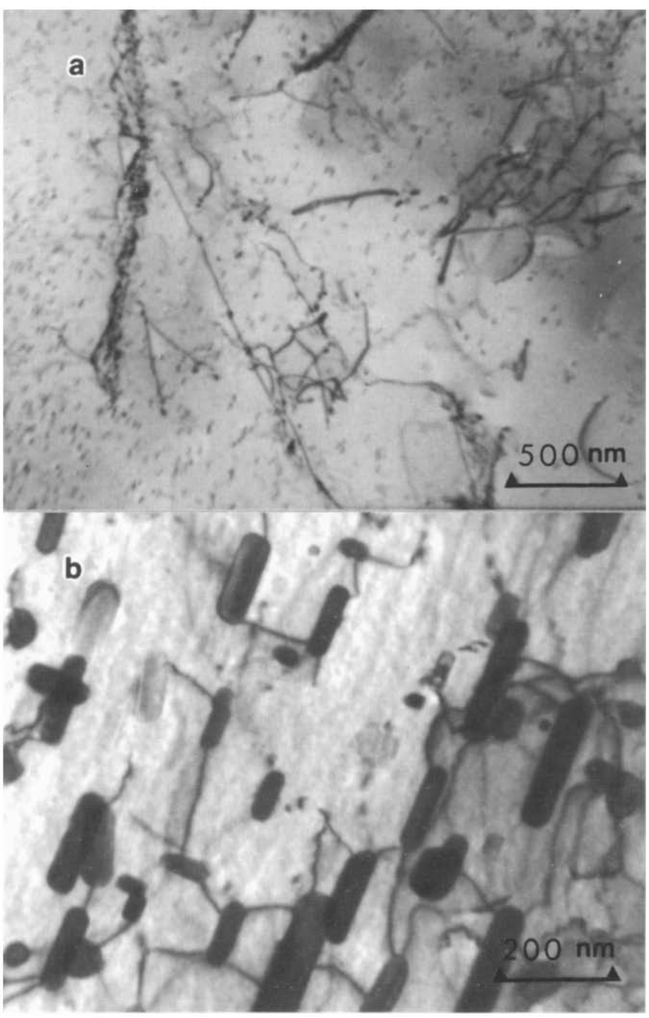

Fig. 8. Typical dislocation structures observed in (a) Al–3.8wt.%Cu aged at 190 °C for 9 h and (b) Al–Cu–Mg aged at 120 °C for 24 h (zone axis ⟨110⟩, diffracting beam ⟨111⟩).

5. Comparison with internal friction measurements

Figure 9(a) shows an elastic creep curve for pure Al, while in Fig. 9(b) we plot the room temperature internal friction Q^{-1} 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. 9(a)), we have derived and reported in Fig. 9(b) 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 ref. 10. The experimental Q^{-1}–ν curve overlaps well with the curve deduced from the static measurements.

Berry and Nowick [8] and, more recently, Parrini and Schaller [11] have systematically analysed the internal friction dependence on thermal treatment for an Al–4%Cu alloy of about the same purity as our Al–3.8%Cu. In particular, we compare internal friction results obtained on a sample aged for 10 h at 203 °C with the anelastic static behaviour that we measured at 22 and 42 °C for a similarly treated sample (Fig. 10). In order to accomplish the transformation, the Zener peak was assumed to be 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 from the activation energies given in ref. 8. The experimental static behaviour of the alloy at room temperature appears to be well matched by the dynamic data measured between 50 and 200 °C.

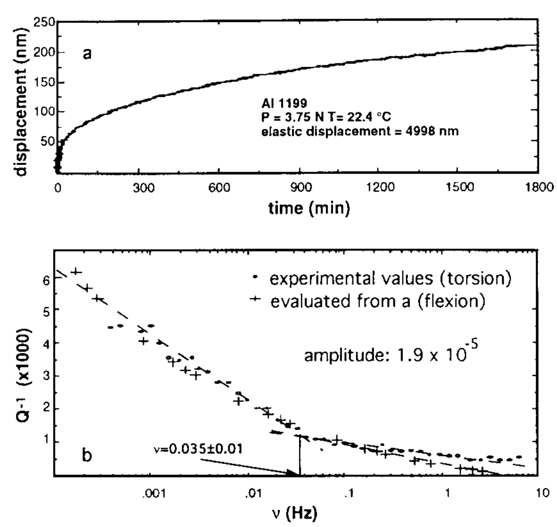

Fig. 9. (a) Anelastic creep and (b) internal friction–frequency curve for pure Al. The experimental values of internal friction evaluated in a forced pendulum are plotted together with the response obtained by transforming the curve in (a).

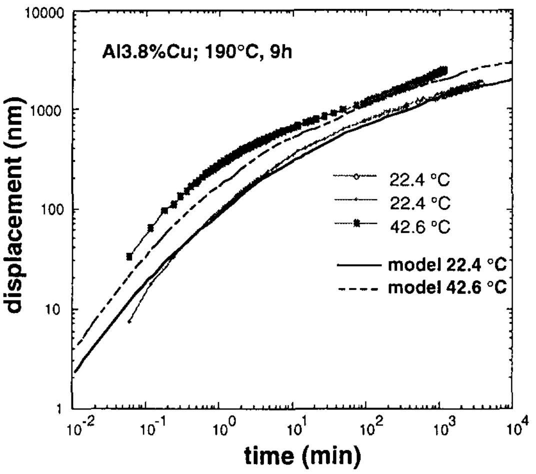

Fig. 10. Anelastic creep of Al–3.8%Cu aged at 190 °C for 9 h. The experimental curves are reported together with the curves obtained from the conversion of the results of ref. 8.

6. Conclusions

The conversion of an internal friction response into a static curve through a deconvolution of the well-known θ' and Zener relaxation peaks shows that the anelastic creep of Al–Cu measured at room temperature is governed by the same mechanisms as are observed at higher temperatures in dynamic measurements.

Unlike the short-time part of the spectrum of binary Al–Cu, which could easily be interpreted in terms of a θ' relaxation mechanism, the short-time peaks for Al–Cu–Mg are more difficult to interpret. The precipitation for the latter alloy is more complex than for Al–Cu, and several concurrent mechanisms may influence its room temperature anelastic creep. The spectra for different aging times become different mainly at the longest relaxation times τ. Several other peaks, observed for τ between 0.1 and 10 min, have a non-monotonic dependence on aging time.

In conclusion, for many application fields (nano-positioning, load cells etc.), high precision measurement of anelastic creep allows the elastic stability of a sample to be controlled at a nanometric level. With respect to dynamic measurements, the method allows reversible and irreversible deformations to be easily distinguished. However, the interpretation of the creep curves through spectral data analysis provides only qualitative information on the form of the spectrum. Internal friction measurements and microstructural observations are important to support the interpretation of the anelastic creep curves.

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