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Anelasticity of ultrafine-grained polycrystalline gold

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Abstract

The internal friction Q^{-1} and resonant frequency f were measured on ultrafine-grained Au specimens prepared by the gas deposition method over the temperature range from 80 to 830 K using a vibrating reed technique (at about 10^2 Hz). The grain size of the specimens was about 30 nm. The modulus defect $\Delta M/M = \Delta(f^2)/f^2$ showed pronounced recovery during annealing at 450–810 K. This recovery can possibly be ascribed to the annealing of lattice dislocations. At higher temperatures three Q^{-1} peaks at around 460, 550 and 750 K were observed. From the behaviours during annealing it is shown that the 460 K peak can tentatively be ascribed to the motion of dislocations with many irregularities, including jogs, and the other two peaks are the ordinary grain boundary peaks. At lower temperatures two Q^{-1} peaks were observed and their possible origins are discussed.

1. Introduction

Ultrafine-grained polycrystalline materials attract much interest because of their unusual physical and mechanical properties [1, 2]. In particular, nanocrystalline materials in which the crystal size D is less than about 10 nm and about 20%–50% of the atoms are in intercrystalline regions are expected to show properties different from both amorphous and ordinary crystalline materials [1]. Therefore their anelastic properties are of great interest. The first complete experimental work on the anelasticity of nanocrystalline materials was reported by Weller *et al.* [3]. They used nanocrystalline Pd specimens ($D \approx 7$ nm) prepared by inert gas condensation of ultrafine Pd particles with subsequent compaction under a pressure of 2 GPa at room temperature (RT). During the first warm-up of an as-prepared specimen they observed a pronounced recovery of elastic modulus centred at around 400 K which was accompanied by a transient internal friction Q^{-1} peak and a subsequent gradual recovery up to 640 K. The amount of recovery in modulus defect $\Delta M/M$ was about 10% in total. They attributed this modulus recovery at around 400 K to an increase in the modulus of grain boundaries M_i associated with interatomic rearrangements in the metastable interfacial structure. From the boundary thickness $d \approx 1$ nm and the relative volume of boundaries $V_i/V_0 \approx 3d/D \approx 40\%$, M_i in the as-prepared specimen was estimated to be about 0.6 M_0, where M_0 is the modulus of crystals.

Recently Akhmadeev *et al.* [4] measured Q^{-1} and $\Delta M/M$ of Cu specimens in the frequency range from 10 Hz to 5 MHz which were heavily deformed at RT by a simple shear using the equichannel angular pressing method with a resulting grain size $D = 200$ nm. They have also observed at around 400 K a stepwise recovery in $\Delta M/M$ of about 10% which was accompanied by a transient Q^{-1} peak. Since the contribution of low M_i to $\Delta M/M$ is negligible in this case ($V_i/V_0 \approx 1.5\%$), they ascribed this recovery to an annihilation of unknown defects in the non-equilibrium boundaries. The interfacial structures may differ for specimens prepared by different methods, but positron lifetime measurements suggest that specimens prepared by the above two methods exhibit a similar structure of the free volumes in the interfaces [5]. Thus the above two interpretations appear to be somewhat contradictory.

Further, recent studies using electron microscopy [6, 7] and X-ray diffraction [8, 9] indicated that the boundary structure of nanocrystalline Pd is essentially the same as that of coarse-grained Pd, so that the presence of ultrafine porosity rather than highly disordered interfacial structures must give the unique character to nanocrystals [6, 8]. In the present work Q^{-1} and $\Delta M/M$ were measured on ultrafine-grained Au (UFG-Au) prepared by the gas deposition method in order to contribute to clarifying the nanocrystalline structure.

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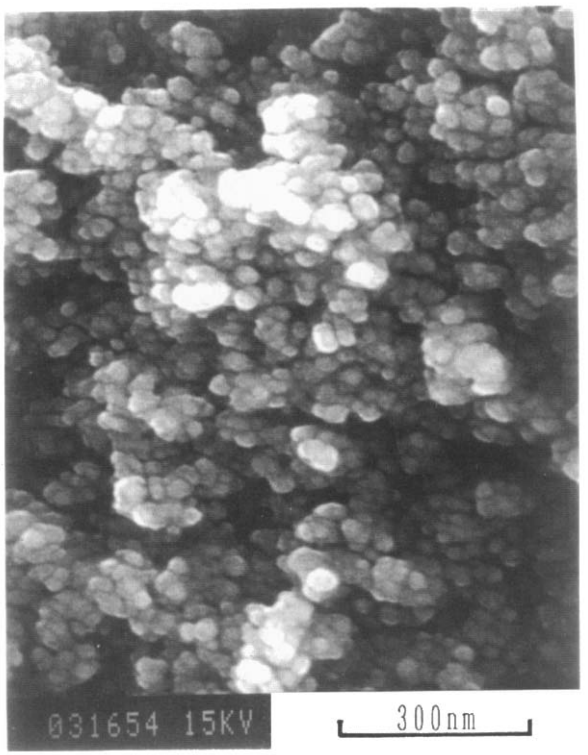

Fig. 1. Scanning electron micrograph of an ultrafine-grained Au specimen prepared by the gas deposition method.

2. Experimental procedures

Specimens were prepared by the gas deposition method [10]. Ultrafine particles of Au (99.99% pure) evaporated in an evaporation chamber with 500 Torr He were introduced into a deposition chamber with 7 Torr He through a fine transfer tube and deposited on a glass substrate at RT in the latter chamber. Then the deposited films were removed from the substrate to form specimens. Typical dimensions of the specimens were 10 μm × 0.4 mm × 20–40 mm. Their grain size was about 30 nm as shown in Fig. 1. The Q^{-1} and resonant frequency f were measured by a vibrating reed technique (at $f \approx 10^2$ Hz) as described in ref. 11 over the temperature range from 80 to 830 K. $\Delta M/M$ is related to f by $\Delta M/M = \Delta(f^2)/f^2$. The measurements were made during linear heating at 1 K min^{-1}.

3. Experimental results and discussion

Examples of the results on UFG-Au are shown in Figs. 2(a) and 2(b), where heating runs were made up to increasingly higher temperatures. A pronounced continuous recovery of $\Delta M/M$ is seen between 450 and 810 K (Fig. 2(a)). At higher temperatures Q^{-1} shows three peaks at around 460, 550 and 750 K (Fig. 2(b)). The 460 K peak decayed during annealing at 450–800 K, the 550 K peak grew and moved to slightly higher

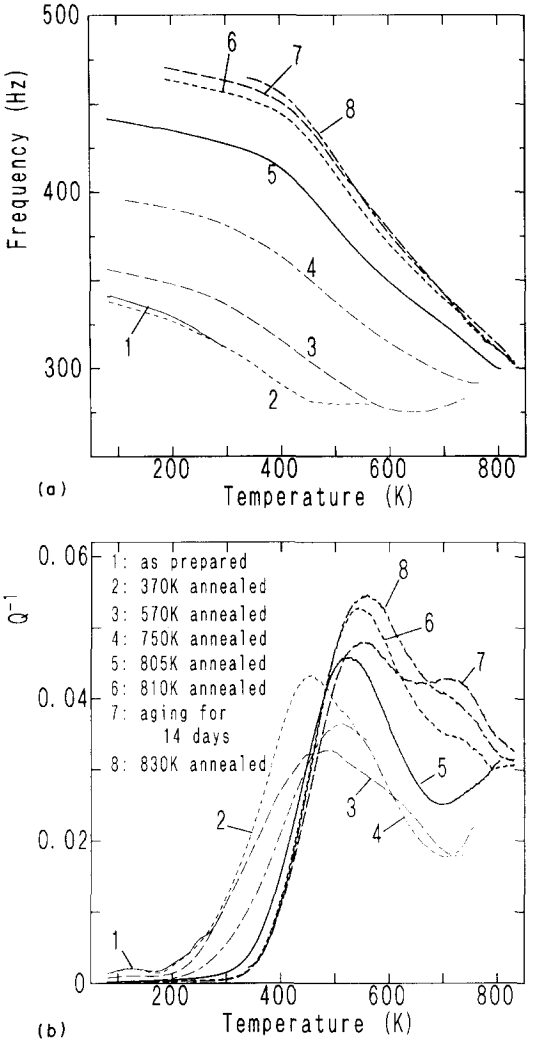

Fig. 2. (a) Resonant frequency f and (b) internal friction Q^{-1} *vs.* temperature observed for a UFG-Au specimen. Each curve was obtained after heating to successively higher temperatures in numerical order as indicated on the figure. The heating rate is 1 K min^{-1}.

temperatures during annealing above 750 K and the 750 K peak appeared after annealing above 805 K. After annealing at 1100 K only the 750 K peak and the background increase at higher temperatures remained but are not shown here.

Since the peak temperatures and behaviours of the 550 and 750 K peaks correspond well with two ordinary grain boundary peaks observed by de Morton and Leak [12], these peaks are probably due to grain boundary sliding without impurities and that modified by impurities respectively [13]. The growth of the 550 K peak would possibly correspond to the atomic rearrangement in and/or around grain boundaries in which metastable boundary structures change to stable ones.

As for the 460 K peak, the grain boundary peak in pure metals consists of two components: the one at lower temperatures is due to dislocations in grains and

the other is due to boundary sliding [14, 15]. Therefore the 460 K peak may tentatively be ascribed to the motion of dislocations with many irregularities, including jogs, which disappear after stabilization of boundaries during annealing at 450–810 K.

Since this specimen bent severely during annealing owing to residual strains, the amount of recovery of $\Delta M/M$ is not reliable. The results shown in Fig. 3 are for a UFG-Au specimen which showed much smaller bending. Here the recovery of $\Delta M/M$ under 450–810 K annealing amounts to more than 30%.

For comparison purposes the results of similar measurements on a coarse-grained Au foil (nominal purity 99.95%, 20 μm thick) deformed at RT are shown in Fig. 4. It is noted that the results after 100% bending are quite similar to those shown in Figs. 2 and 3, except for a much smaller change in Q^{-1} in Fig. 4, while the results after 60% rolling are very similar to those reported in ref. 4. The recovery of $\Delta M/M$ in the coarse-grained specimen can usually be explained by a recovery of mobile lattice dislocations. Even in ultrafine-grained specimens, if each grain of diameter D contains a dislocation line of free length D, under the applied stress σ the mean displacement of the dislocation given by $\bar{y} \approx \sigma D^2/6Gb$ contributes to a dislocation strain $\epsilon_d = \bar{y}b/D^2 \approx \epsilon_0/6$. Here G, b and ϵ_0 are the shear modulus, Burger's vector and elastic strain respectively. Then $\Delta M/M = \Delta G/G = \epsilon_d/(\epsilon_0 + \epsilon_d) \approx 14\%$ and this is independent of the grain diameter D. Therefore two dislocation lines in each grain can explain $\Delta M/M \approx 30\%$. Now, comparing the results on the coarse-grained specimen with those on the UFG-Au specimens, it appears that the large recovery of $\Delta M/M$ in UFG-Au specimens is most likely to be explained by the recovery of dislocations in grains which are in a metastable state in the as-prepared specimens. On the other hand, this large recovery in $\Delta M/M$ is impossible to explain by the recovery of low M_i as reported in ref. 3.

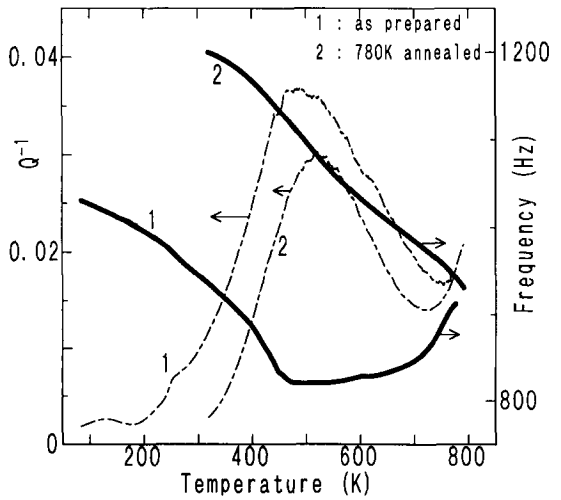

Fig. 3. Similar to Fig. 2 but for the other UFG-Au specimen which showed a little bending during heating and was directly heated to 780 K.

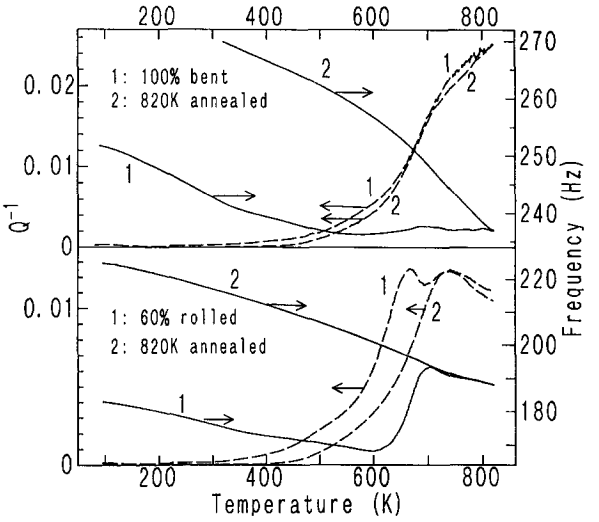

Fig. 4. Resonant frequency f and internal friction Q^{-1} *vs.* temperature observed for a coarse-grained Au foil (99.95% pure) after deformation by bending at RT (top) and after additional deformation by rolling at RT (bottom).

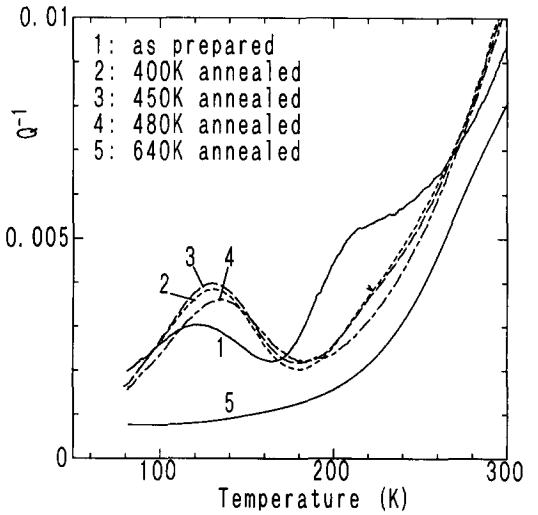

Fig. 5. Similar to Fig. 2 but showing only the lower temperature region.

Finally, the Q^{-1} spectrum below 300 K in UFG-Au should be mentioned briefly. As shown in Fig. 5, the peaks at 120–130 and 210 K (about 300 Hz) are noted whose behaviours are similar to the Bordoni peak [16] and the P_1 peak (relaxation peak associated with point defect–dislocation interaction) [17] respectively. However, after the complete disappearance of the peaks upon annealing at 810 K, these peaks reappeared again after aging for 2 weeks at RT (not shown here) and at the same time the Q^{-1} spectrum at high temperatures also showed a peculiar change as seen in Fig. 2(b). The reason for this peculiar effect is not known but,

because of the porosity of the specimens, a hydrogen absorption is suspected.

In the present work we have not yet been successful in observing grain growth or other property changes and further work is now in progress.

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