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Mechanical Spectroscopy in Nitrogen Implanted Copper

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Abstract. The effects of nitrogen irradiation employing a plasma focus device on high purity polycrystalline copper are shown. It should be pointed out, that the effects of implantation process depend strongly of the previous thermal state of the sample. In fact, a new damping peak at approximately 750 K appears in irradiated samples which were previously annealed during 1 hour at 1073 K, but neither new peak at 750 K was measured in samples previously annealed during 5 and 15 hours. However other new peak appears at 1000 K. Moreover in the first case non hysteresis effects between a run up and down in temperature were found in both damping and modulus, but in the second one strong hysteresis effects appeared.

1. INTRODUCTION

The nitrogen implanted samples have shown a new damping peak at approximately 750 K, absent in the unimplanted ones, and also an increase in the elastic modulus which depend on the final fluence of implantation [1]. These changes produced by the nitrogen implantation were stables during a run up and down in temperature, i. e. non hysteresis effects were detected. On the contrary, the argon implanted samples have shown also an increase in the elastic modulus and an anomalous behaviour in it. It consist in a jump toward higher values during the run up in temperature, which disappears after 48 hours of implantation [2]. This jump decreases for a larger annealing time of sample previous to implantation processes [3].

In this work new measurements of damping were performed in polycrystalline copper samples annealed during 5 and 15 hours at 1073 K under high vacuum which were nitrogen implanted with different ions fluences. These new results are compared with the already reported ones.

2. EXPERIMENTAL PROCEDURE

Mechanical spectroscopy measurements were performed in an inverted torsion pendulum at variable moment of inertia which operates at low frequency from 0.2 Hz up to 40 Hz [4]. The pendulum works at low and high temperatures under high vacuum and it is driven by a PCL-812 Card [4]. The measurements were carried out in free decay, with a temperature increase rate of 0.5 K/min. Decreasing temperature was made at the same rate. The maximum shear strain on the sample was 4×10^{-5} . The damping values were considered as amplitude independent owing to the degree of dependence do not obstruct the subsequent spectra analysis. The measured damping spectra in unirradiated samples did not present hysteresis effects. Moreover, increasing the heating rate toward higher values the damping spectra, both of unirradiated and irradiated samples, were rigidly shifted toward higher temperatures. The natural frequency at room temperature in the test was 0.6 s^{-1} .

The implantations were carried out with a pulsed ion implanter, which provides ion beams of short time duration between 30 ns - 500 ns, with a continuous energy spectrum following the spectral law $dN/dE \propto E^{-3/4}$ where N is the number of ions with energy E [5]. In a beam it is possible to find ions with energy from 20 KeV to several MeV. The final fluence, FF, for nitrogen, was reached after a predetermined

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number n of single pulses, ff ($FF = n \times ff$). As a consequence of the pulsed nature, when a beam interacts with the target in an implantation processes, it induces strong thermal effects which have to be added to a pure ion implantation process [6].

The samples were made with polycrystalline high purity electrolytical copper 99.99% in sheets of $30 \times 4.5 \times 0.3 \text{ mm}^3$. The thermal treatments were carried out under high vacuum during 1, 5 and 15 hours at 1073 K, previously to irradiations. The status of checked samples is written in Table. 1.

Sample	Annealing Condition	Implantation Condition $FF(\text{ions/cm}^2)$
1	1073 K during 1 hour	NO
1''	"	1×10^{15}
1'''	"	2×10^{15}
2	1073 K during 5 hours	NO
2''	"	1×10^{15}
3	1073 K during 15 hours	NO
3'	"	0.5×10^{15}
3''	"	1×10^{15}
3'''	"	2×10^{15}

Table 1 : Samples Status

3. RESULTS

Figure 1 shows the damping spectra, F , for the set of samples number 1, i. e. samples 1, 1'' and 1'''. The damping for the unimplanted sample shows the low and intermediate temperature grain boundary peaks of the copper (called LTP and ITP) which appear at approximately 550 K and 720 K (at 1 Hz) respectively [8]. In the implanted samples 1'' appears the already mentioned new peak at approximately 750 K, absent in the unirradiated ones [1]. Furthermore, the spectrum for the irradiated samples 1''' was strongly increased.

The elastic modulus increases about 10% and 80% for irradiated samples 1'' and 1''' respectively with respect to unirradiated ones.

The behaviour during the cooling of the damping did not present hysteresis effects. The modulus hysteresis effects during the cooling are smaller and do not recover the values of unirradiated samples [1].

Figure 1: Damping spectra for the set of samples number 1, full (1), short (1'') and alt dashed (1''').

Figure 2: Damping spectra for a set of samples number 2. Left axis : run up in temperature. Right axis : run down. Full line (2), dashed line (2'').

The damping behaviour as a function of temperature for the set of samples number 2 is plotted in figure 2. The LTP and ITP for unirradiated samples appear overlapped [7]. It should be pointed out that

the new peak owing to irradiation processes did not appear. Moreover, the damping decreases within the temperatures range around 750 K. Nevertheless, another new peak appears at approximately 1000 K which is absent in the unirradiated ones. Furthermore, strong hysteresis effects in the damping appear during the cooling, as can be seen also from figure 2. The elastic modulus also presented an hysteresis behaviour. The modulus values decrease during the cooling reaching the values of the unirradiated samples.

Figure 3 shows the behaviour of measured damping for the set of samples number 3. It is similar to the behaviour of samples 2. In fact, the damping spectra in the range of temperature around 750 K decrease. Furthermore, other new peak at approximately 1000 K, absent in the unimplanted ones, appears. These effects are larger for a larger irradiation fluence. Moreover strong hysteresis effects in the damping appear during the cooling as can be seen also in the figure 3 by shifting the base line. Besides this, the elastic modulus also presents an hysteresis behaviour during the cooling, where these values reach to ones in the unirradiated samples.

Figure 3: Damping spectra for a set of samples number 3 during the run up in temperature plotted in the ground axis. Spectra during the run down plotted by shifting the base line. Full line (3), dotted (3'), dashed (3'') and alt dashed line (3''')

The experimental results can be summarised as follows : (a) in samples annealed during 1 hour. (a.1) the peak appears in irradiated samples at 750 K, (a.2) the damping spectrum is strongly increased for larger ions fluences, (a.3) non hysteresis effects were detected in the damping spectrum between a run up and down in temperature, (a.4) the modulus increases in implanted samples and it does not present important hysteresis effects. (b) in samples annealed during 5 and 15 hours, (b.1) the peak at 750 K does not appear and the damping values around 750 K decrease for a larger fluence, (b.2)) other peak appears at approximately 1000 K absent in the unirradiated samples, (b.3) damping hysteresis effects appear between a run up and down in temperature. (b.4) the modulus increases in implanted samples and it recovers after having reached 1000 K.

4. DISCUSSION

The already proposed mechanism which could control the irradiation peak in samples annealed 1 hour was the introduction of impurities which modificate the grain boundary sliding rate. Such an impurity could be nitrogen atomic molecules of N-O, N-N, N-N-N or precipitates [1]. The behaviour shown by the damping spectra of irradiated samples 2 and 3, i. e. the reduction in the peak height of the ITP and the arising of a new peak at higher temperatures, could be also related to the introduction of defects by the implantation processes which reduce the grain boundary sliding rate [8]. The strong hysteresis effects

during cooling could be related to precipitation processes that eliminate the sliding capacity. The precipitates could be some kind of compound generated during the implantation processes, but this point is controversial and it will be explored in detail in further works. The behaviour of modulus does not show an increase related to precipitation during cooling, owing to that the effects of the recuperation of irradiated structure could be greater than the precipitation ones. The softening during cooling which could be related to recovery of irradiated structure after having reached 1050 K is in agreement with the results reported on the recovery of radiation damage in copper [9]. On the contrary the stability at high temperatures in the modulus behaviour without recovery, in the set of samples number 1 must be even more studied.

It should be pointed out that, the following points must be also considered carefully for explaining the damping spectrum and modulus behaviour in nitrogen implanted samples. 1) the effect of the argon implantation on the damping in copper. In fact, the argon implanted samples show a damping peak at 840 K which has an inverse modulus relaxation behaviour, i. e. the modulus increases. Furthermore the peak and the jump in the modulus are unstable in the time [2]. 2) The strong increase of the damping spectrum in the set of samples 1 for larger fluences. Therefore, another mechanism could be operating (joined to the grain boundary structure) in the generation of the peak at 750 K produced by the implantation processes. This could be, the dragging of point defects which follow the dislocation line motion [10, 11]. The pinning of dislocations also should be suggested as other cooperative mechanism owing to the decrease in the damping values in samples 2 and 3 for larger ions fluences [12].

Resuming, a cooperative mechanism between the grain boundary structure and the dragging and pinning of dislocations, where the agglomerates were generated by the implantation processes, could be the physical mechanism which controls the peak at 750K in implanted samples. More effort is required for explaining this phenomenon, beginning for a complete characterisation of samples after implantation processes is necessary for interpreting this effects.

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