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Phase transitions in copper–silver alloys under high pressure torsion

The influence of high pressure torsion (HPT) on the formation and decomposition of solid solutions in the copper–silver system has been studied. The investigated Cu-8 wt.% Ag alloy was annealed at two different temperatures, 500 and 650 °C, and quenched. The samples consisted of (Cu) solid solution in the matrix with (Ag) precipitates. During HPT a steady-state value of torsion torque was reached after about 1.5 anvil rotations. After HPT (5 anvil rotations) the composition of the (Cu) solid solution in both samples had become equal. In other words, the concentration of silver in the (Cu) matrix annealed at 650 °C decreased and in the sample annealed at 500 °C increased. Moreover, a similar process took place in (Ag) precipitates as well. The concentration of copper in (Ag) particles in the sample annealed at 650 °C decreased and in the sample annealed at 500 °C increased. Thus, the composition of (Cu) and (Ag) solid solutions reached at steady-state during HPT does not depend on that before HPT. The composition of the (Cu) and (Ag) solid solutions after HPT is as high as if the samples were annealed at a certain intermediate temperature about 600 ± 20 °C.

Keywords: Phase transitions; Severe plastic deformation; High pressure torsion; Decomposition; Precipitation

1. Introduction

The processes occurring in materials under the action of severe plastic deformation (SPD) attract continuous and even the continuously growing interest of materials science researchers [1, 2]. In particular, very important is the evolution of two-phase polycrystals under SPD, and especially in conditions of pure shear during high pressure torsion (HPT) [3–11]. For a long time it was broadly accepted opinion that SPD always leads to the grain refinement, fragmentation of structure, dissolution of precipitates and even amorphization [12–40]. Later it was observed that the opposite process can also take place during SPD. Namely, a supersaturated solid solution can decompose under the action of SPD and fine particles of a second phase can precipitate from a matrix during this process [41–44]. In further investigations it has been discovered that the enrichment of a solid solution (with dissolution of precipitates), on the one hand, and the decomposition of a supersaturated solid solution (with precipita-

tion of fine particles), on the other hand, can take place simultaneously in the same system [45, 46]. Moreover, these processes compete with each other and – after a certain HPT strain – they come into dynamic equilibrium [47]. In this dynamic equilibrium a certain steady-state concentration is reached in the matrix solid solution [48].

The SPD-driven decomposition of a supersaturated solid solution was observed for the first time in the Al–Zn alloys [41, 49–52]. The competition between dissolution and precipitation has been observed in several Cu-based alloys [48]. However, the important question remains open to the present time: how does SPD influence the composition of precipitates of a second phase? Does it also change, and if yes, how does it change? In order to answer this question we will use the well studied Cu–Ag system where the composition of (Ag) precipitates in (Cu) matrix can vary from 0 to 8.5 wt.% Cu [53]. Therefore, the maximal concentration of copper in (Ag) precipitates is high, and its changes during HPT are measurable.

2. Experimental procedure

A Cu-8 wt.% Ag alloy was investigated in this work. This concentration is close to the maximal solubility of Ag in Cu. Therefore, the (Ag) precipitates should exist in the samples after all heat treatments. They also should remain in the sample after HPT treatments. It allowed us to be able to observe the Ag X-rays diffraction (XRD) peaks and to measure the lattice parameter not only in the Cu matrix but also in the (Ag) precipitates. The alloy was prepared from high purity 5N components by means of vacuum induction melting. The melt was poured under vacuum into a water-cooled cylindrical copper crucible of 10 mm diameter. For HPT processing, 0.6 mm thick discs were cut from the as-cast ingot, then ground and chemically etched. The discs were then sealed into evacuated silica ampoules with a residual pressure of approximately 4×10^{-4} Pa at room temperature. The sealed ampoules were annealed at 500 °C for 600 h and at 650 °C for 100 h. After annealing, the ampoules were quenched in water. The accuracy of the annealing temperature was ± 3 °C. The annealed and quenched Cu–Ag discs were subjected to HPT in a Bridgman anvil type unit (room temperature, pressure 5 GPa, 5 torsions, 1 rotation-per-minute) using a custom-built computer controlled HPT device (W. Klement GmbH, Lang, Austria). The temperature of the sample increases during HPT only negligibly, up to

about 40–50 °C. After HPT, the central (low-deformed) part of each disc (about 3 mm in diameter) was excluded from further investigations. The samples for structural investigations were cut from the deformed discs at a distance of 4–5 mm from the sample centre. Scanning electron microscopy (SEM) investigations were carried out in a Tescan Vega TS5130 MM microscope equipped with LINK energy-dispersive spectrometer and on a Philips XL30 scanning microscope equipped with a LINK ISIS energy-dispersive spectrometer produced by Oxford Instruments. XRD data were obtained on a Siemens diffractometer (Co- K_{α} radiation). Grain (crystallite) size was estimated by the XRD line broadening and using the Scherer formula.

3. Results and discussion

Figure 1a and b shows SEM micrographs of the structure of Cu-8 wt.% Ag alloy annealed at 500 °C for 600 h (a) and at 650 °C for 100 h (b) before HPT. (Cu) matrix appears dark,

(Ag) precipitates appear bright. (Cu) matrix contains the uniformly distributed (Ag) particles. Grain size in (Cu) is about 20–40 μm . The size of (Ag) particles is about 2–5 μm . After HPT, the (Ag) particles become fragmented and very fine (Fig. 1c and d). They form a layered structure. The layers are parallel to the plane of rotating HPT anvils.

Figure 2 shows the XRD patterns for the Cu-8 wt.% Ag alloy annealed at 500 °C for 600 h before (bottom curve) and after (top curve) HPT with 1 rpm, 5 rot. Dotted lines permit comparison of the positions of (Ag) and (Cu) peaks after HPT with those before HPT. The (Ag) and (Cu) XRD peaks are broadened after HPT, demonstrating the strong grain refinement after HPT. The grain size can be roughly estimated as 150–300 nm for (Cu) and 20–50 nm for (Ag). After HPT the (Cu) peaks move to the lower values of diffraction angles 2θ . This corresponds to an increase in the Cu lattice parameter. Conversely, the (Ag) peaks move after HPT to the higher values of 2θ , corresponding to a decrease in the Ag lattice parameter.

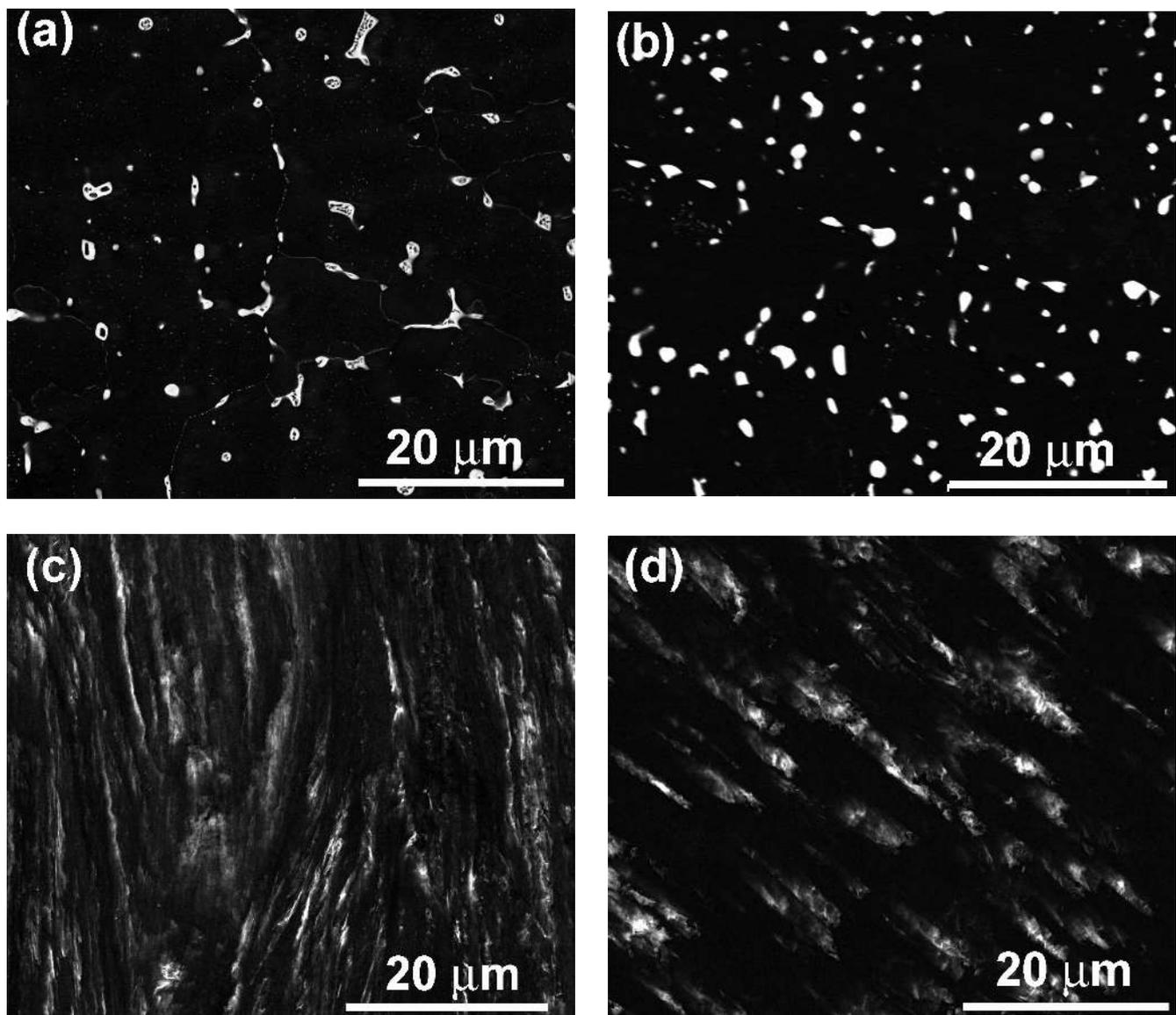


Fig. 1. SEM micrographs showing the structure of Cu-8 wt.% Ag alloy annealed at (a) 500 °C for 600 h, and (b) at 650 °C for 100 h before HPT (a,b) and after HPT (c) and (d), respectively. (Cu) matrix appears dark, (Ag) precipitates appear bright.

Figure 3a shows the dependence of the (Cu) lattice parameter with the concentration of silver. Small filled symbols are the values taken from the literature [54–57]. They can be very well approximated by the linear dependence (straight solid line) known as Vegard’s law. Vegard’s law is valid for nearly ideal solid solutions [58, 59]. Thus, the (Cu) lattice parameter increases with increasing content of silver. Big open circle and diamonds correspond to the (Cu) lattice parameter after annealing at 500 °C, 600 h and 650 °C, 100 h, respectively. Thus, the big open symbols show the (Cu) lattice parameter before HPT. The big plus and cross show the (Cu) lattice parameter of the same samples after HPT. The curved arrows show how the HPT influences the (Cu) lattice parameter. It can be seen that HPT increases the lattice parameter of the sample annealed at 500 °C and decreases that of the sample annealed at 650 °C. The resulting values of lattice parameter “come together” to the point between two starting points (before HPT). Using Vegard’s law has permitted us to estimate the concentration in (Cu) solid solutions from the measured lattice parameter. The resulting values “come together” to the point at ≈ 1.73 at.% Ag between two starting points of ≈ 1.25 and ≈ 1.95 at.% Ag, respectively.

Figure 3b shows the dependence of (Ag) lattice parameter on the concentration of silver. Small filled symbols are the values taken from the literature [55, 57, 60]. They also can be very well approximated by Vegard’s law (straight solid line). The (Ag) lattice parameter decreases with increasing content of copper. Big open circle and diamond correspond to the (Ag) lattice parameter after annealing at 500 °C, 600 h and 650 °C, 100 h, respectively. The big plus and cross show the (Ag) lattice parameter of the same samples after HPT. The curved arrows show how the HPT influences the (Ag) lattice parameter. It can be seen that HPT increases the lattice parameter of the sample annealed at 500 °C and decreases that of the sample annealed at 650 °C. The resulting values move towards each other to the points between two starting points (before HPT). Also

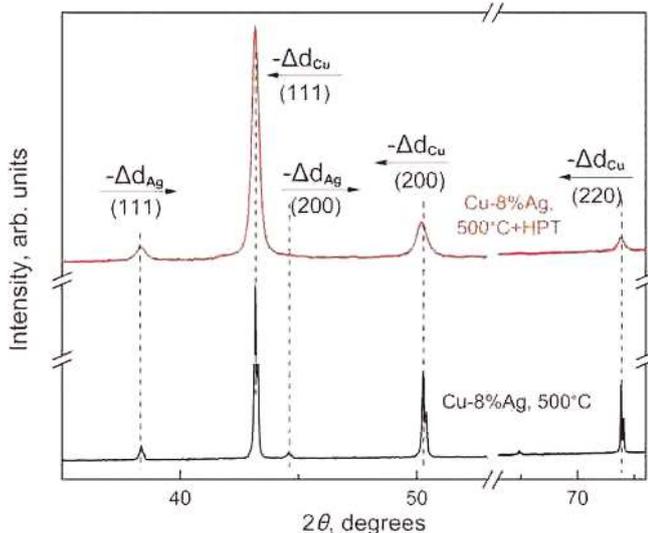


Fig. 2. XRD patterns for the Cu-8 wt.% Ag alloy annealed at 500 °C for 600 h before (bottom curve) and after (top curve) HPT with 1 rpm, 5 rot. Dotted lines allow comparing the position of (Ag) and (Cu) peaks after HPT with those before HPT.

in this case, using Vegard’s law has permitted us to estimate the concentration in (Ag) solid solutions from the measured lattice parameter. The starting points were at ≈ 3.1 and ≈ 6 at.% Cu. The concentrations after HPT were ≈ 4.3 and ≈ 5.3 at.% Cu, respectively.

Figure 4 shows the Ag–Cu phase diagram [53]. Open circles on the solvus lines show the concentrations in (Cu) and (Ag) solid solutions before HPT. Open squares on the respective solvus lines show the concentrations in (Cu) and (Ag) solid solutions after HPT. Curved arrows show how

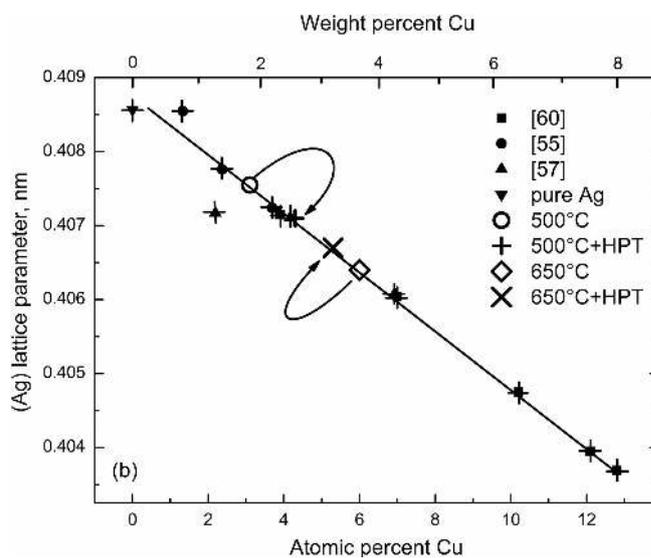
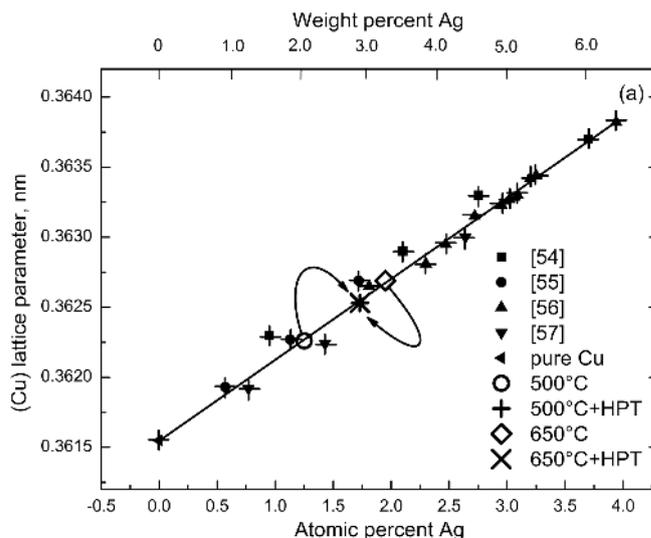


Fig. 3. Dependence of (a) Cu, and (b) Ag lattice parameter on the concentration of silver and copper, respectively. Small filled symbols are the values taken from the literature [54–57, 60]. They are approximated by the straight solid line (Vegard’s law [58, 59]). Big open circle and diamond correspond to the (Cu) and (Ag) lattice parameter after annealing at 500 °C, 600 h and 650 °C, 100 h, respectively. The big plus and cross show the (Cu) and (Ag) lattice parameter of the same samples after HPT. The dimension of big symbols corresponds to the respective error bars. The curved arrows show how the HPT influences the (Cu) and (Ag) lattice parameter.

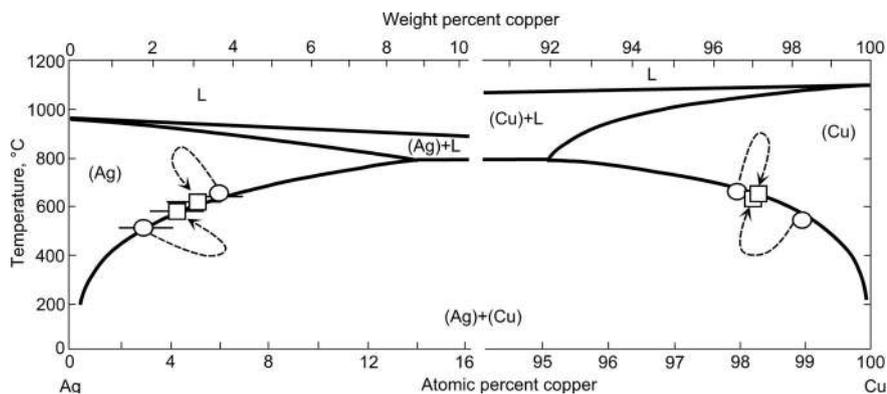


Fig. 4. Ag–Cu phase diagram [53]. Open circles show the concentrations in (Cu) and (Ag) solid solutions before HPT. Open squares show the concentrations in (Cu) and (Ag) solid solutions after HPT. Curved arrows show how the HPT influences the concentrations in (Cu) and (Ag) solid solutions.

the HPT influences the concentrations in (Cu) and (Ag) solid solutions. It can be seen that the concentration of silver in the (Cu) matrix of the sample annealed at 650 °C decreased and in the sample annealed at 500 °C increased. Moreover, a similar process also took place in (Ag) precipitates. The concentration of copper in (Ag) particles in the sample annealed at 650 °C decreased and in the sample annealed at 500 °C increased. Thus, the composition of (Cu) and (Ag) solid solutions reached in the steady-state during HPT does not depend on those before HPT. The composition of (Cu) and (Ag) solid solutions after HPT is as high as if the samples were annealed at a certain intermediate temperature about 600 ± 20 °C.

This effect is not due to the adiabatic temperature rise during HPT. Indeed, the heating of the sample during HPT is not adiabatic. It is easy to understand because the heat is released during HPT in a very small sample with the mass of few grams. The temperature only negligibly increases during HPT. Direct measurements with a thermocouple demonstrated the temperature rises only 1 K for Sn, 3 K for Al, 8 K for Cu and 12 K for Ti [61]. The flat and small HPT sample is surrounded by the cold and heavy equipment parts with a mass over 100 kg and released heat dissipates immediately. Moreover, if the temperature of HPT equipment is increased with a special furnace, the resulted structure of samples HPT-treated at 100 °C or even 50 °C is very different from that after treatment at room temperature [62, 63]. This is further proof of the fact that the temperature only negligibly increases during HPT.

The dynamic equilibrium during HPT leading to the saturation of various properties and structural features such as dislocation density [64, 65], microstrain [66], grain size [65, 67–69], amount of ω -phase in titanium alloys [70], hardness [69, 71–73], electrical conductivity [74] has been observed in numerous works. Moreover, the saturation of grain size also has been observed for other SPD modes such as ball milling, equal channel angular pressing, accumulative roll bonding etc. [48]. It was interesting to compare, for example, how the grain size in the same material (copper) depends on the SPD mode [48].

In our case we see how the concentration in (Cu) and (Ag) solid solutions reaches dynamic equilibrium during HPT. Moreover, the steady state concentration of silver in the (Cu) solid solutions and that of copper in (Ag) solid solution “forgets” the starting one. Obviously, it is only the function of the straining process (like pressure, temperature and strain rate in case of HPT). In the dynamic equilibrium the rate of strain-driven defect production becomes equal to the

rate of diffusion-like defect annihilation (or relaxation). The high defect concentration in the steady state “emulates” the temperature increase. It is well known, for example, that the equilibrium concentration of vacancies grows exponentially with increasing temperature. The high concentration of defects in the steady state ensures the increased diffusion-like mass transfer across the interphase boundaries between (Cu) matrix and (Ag) precipitates. As a result, the steady state is established not only for the composition of (Cu) matrix but also for that in the (Ag) precipitates.

Previously, we have observed in Cu-based alloys that the decomposition of supersaturated solid solution (and respective formation of precipitates) competes during HPT with dissolution of particles of a second phase (and respective increase of concentration in a matrix) [45–48]. Both decomposition and dissolution proceed very quickly [45–48]. The estimated equivalent diffusion coefficients of this diffusion-like mass transfer are several orders of magnitude higher than the coefficient of conventional volume diffusion extrapolated to HPT temperature of 300 K. This acceleration is especially astonishing because the applied pressure always decreases the rate of diffusion-controlled processes [75, 76]. The resulting steady-state composition does not depend on the composition of a matrix solid solution in the initial state before HPT. A similar phenomenon was observed for the (Cu) matrix here as well. However, we observed for the first time the changes of copper-containing Ag-based solid solution in the (Ag) precipitates. It is similar to that in (Cu) matrix. Namely, the high concentration of copper decreased during HPT, the low one increased, and a certain intermediate composition of (Ag) particles formed.

4. Conclusions

Two opposite phase transitions proceed simultaneously and compete with each other during severe plastic deformation by the high pressure torsion of two-phase Cu-based alloys. These two processes are the decomposition of supersaturated solid solution (and respective formation of precipitates) and simultaneous dissolution of particles of a second phase (and respective increase in concentration in a matrix). We observed for the first time similar competition not only in the (Cu) matrix but also in copper-containing Ag-based solid solutions in the (Ag) precipitates. It is similar to that in (Cu) matrix. Namely, the high concentration of copper decreased during HPT, the low one increased, and a certain intermediate composition of (Ag) particles formed.

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