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Hardening Mechanisms of Metals and Alloys Produced by SPD

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Abstract. Strengthening is a complex process involving such basic mechanisms as dislocation accumulation (work hardening), Hall-Petch hardening due to grain refinement, solid solution hardening and precipitation hardening in various combinations. The contribution of different mechanisms into resultant strength can vary significantly depending on chemical composition and processing. The purpose of the present work is to explore the significance of SPD for hardening and to clarify the role of different strengthening mechanisms. The model Au-based system was employed using pure Au, single phase solid solution Au-25Ag and precipitation hardenable Au-12.5Ag-12.5Cu (in mass %) alloy subjected to ECAP. The additive character of different strengthening mechanisms is clearly demonstrated. The extremely high strength exceeding 1 GPa is achieved in the Au-Ag-Cu after ECAP followed by aging, which has never been attained in conventional processing schemes.

Introduction

Pure (24 carats) gold has a quite low strength and hardness which impose significant limitations in design of jewelry art works and other commercial products. It has been demonstrated recently that utilizing the equal-channel angular pressing (ECAP) is quite effective for substantial improvement (by a factor of 3 or more) of strength in pure gold and its alloys [1]. In the present brief communication we extend the discussion on the mechanical properties of metals produced by severe plastic deformation. The accent is placed on the mechanisms of strengthening and the role of different metallurgical factors in strengthening. In the present paper, gold is considered as a model material, although this work was initially motivated by a challenging task – development of high strength Au-based alloys for jewelry.

Pure 4N gold (K24), dilute solid solution Au-0.5Ag and the K18 gold alloys such as Au-25Ag – the single phase solid solution and Au-12.5Ag-12.5Cu (all compositions are in mass %) – the precipitation hardenable alloy were explored to shed light on the role of SPD in the enhancement of properties of materials when different strengthening mechanisms contribute to a resultant strength. Among these mechanisms one should list the followings. (1) The dislocation hardening associated with dislocation storage in the grain interior. (2) Hall-Petch or grain boundary (GB) hardening which is associated with grain fragmentation and with the barrier effect of GBs for dislocation movement. (3) Strengthening via texture formation by varying the fraction of grains with "hard" and "soft" crystallographic orientations. These three mechanisms operate in all ECAP materials involved. Furthermore, we should list the following specific contributions. (4) The effect of the short order, which becomes pronounced in dilute solution while the SFE remains practically unchanged (Au-0.5Ag). (5) Solid solution strengthening dominating in alloys with a high concentration of solvents (Au-25Ag) and (6) particle strengthening in multiphase systems (Au-12.5Ag-12.5Cu). Hence, the materials employed in the present study are supposed to involve all these mechanisms in various combinations, which is useful to clarify the role of individual constituent mechanisms in the resultant properties of UFG SPD metals.

Experimental

The ECAP has been performed at room temperature, using a die-set with sharp corners between two rectangular channels of 10×10 mm^2 cross-section intersecting at 90°. Up to four pressings via route Bc (with 90° rotation of the billet between subsequent pressings) have been carried out, i.e. the total strain intensity in the billet reached $\varepsilon_i \approx 4.60$. After each path through the die, the Vickers microhardness HV was measured at the central part of the billet on the flow and bottom planes using a dynamic ultra-hardness tester Shimadzu DUH-1.0 at 10 g load and 15 s exposure time. The average of 5-10 measurements was calculated. Tensile tests were carried out on a screw driven testing machine at a nominal strain rate 1×10^{-4} s^{-1}. The Au-12.5Ag-12.5Cu alloys were quenched from 600°C (40 min) into water before ECAP. The peak aging conditions were found experimentally in series of heat treatments of as-ECAP specimens at different temperatures between 250 and 350°C.

Results and Discussion

The results of microhardness measurements are shown in Figs.1 and 2. The hardness after ECAP increased remarkably for all materials, as was expected. One can notice however, that the degree of strengthening via SPD is different for different chemical compositions of alloys. Apparently, the effect of ECAP on pure Au and its dilute solid solution Au-0.5Ag is practically the same, indicating that the short order strengthening is negligible when compared to the effect of SPD. The strengthening of K18 Au-25Ag is particularly pronounced and the effect of ECAP is most vivid. The contribution of different mechanisms, which were listed in the introduction, can be easily seen and compared in Fig.1. It is interesting to notice that the effect of ECAP is the same for two K18 alloys - Au-25Ag and Au-12.5Ag-12.5Cu, so that the difference in strength or hardness between these alloys is attributed entirely to the effect of solid solution strengthening, which is surely more pronounced in the AuAgCu alloy. The hardness of the latter can be enhanced remarkably after ECAP via particle strengthening.

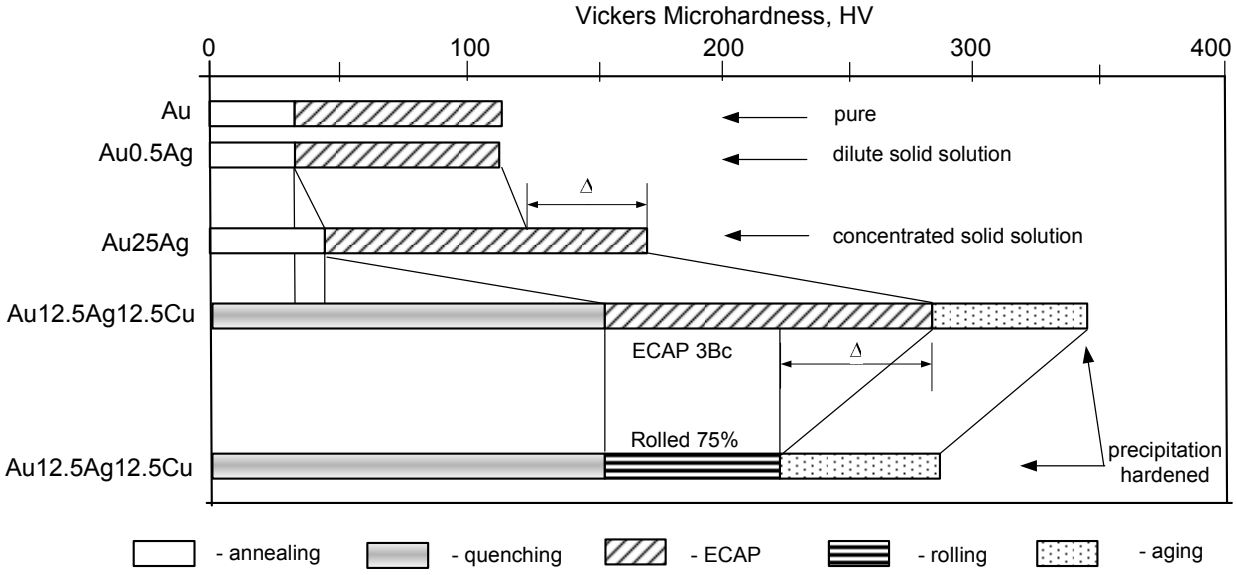

Fig.1. Stack-bar chart illustrating schematically the contribution of different mechanisms to the hardness

Assuming a liner relation, which often exits indeed, between the hardness and strength as $\sigma_{UTS} \approx 3HV$, for example, the power law can be applied to describe the hardening during ECAP in the same way as it is used commonly for describing the monotonic stress-stress curve

$$HV = HV_0 + K\varepsilon_T^n \qquad (1)$$

where ε_T is the true strain and HV_0, K and n are materials properties. As shown in Fig.2, the power low (1) fits the experimental data quite well at least as long as the number of pressings is small and the dominant mechanism of strengthening remains to be the dislocation accumulation and the subs-structure formation. We say that "the number of small" when the structure is not yet converted into the granular type with prevailing high-angle grain boundaries. There has been a plenty of evidence in the literature that the high-angle grain boundaries tend to form in f.c.c. metals at considerably higher strains (number of ECA-passes) than we imposed in the present work [2]. Surely, the number of passes which is required to form the high-angle grain boundaries is different for different materials, being dependent on various metallurgical factors such as the stacking fault energy, for instance. It is worth noticing that the strain hardening exponent n is the same for both K18 alloys, i.e. strengthening in Au-25Ag and AuAgCu occurs in the essentially same way despite considerably different properties of solid solutions. As a result, we obtain the similar hardening effect after ECAP for both compositions, Fig.1, and a higher strength of AuAgCu alloy is entirely associated with the higher strength of the supersaturated solid solution after quenching if compared with the fully-annealed state of Au-25Ag.

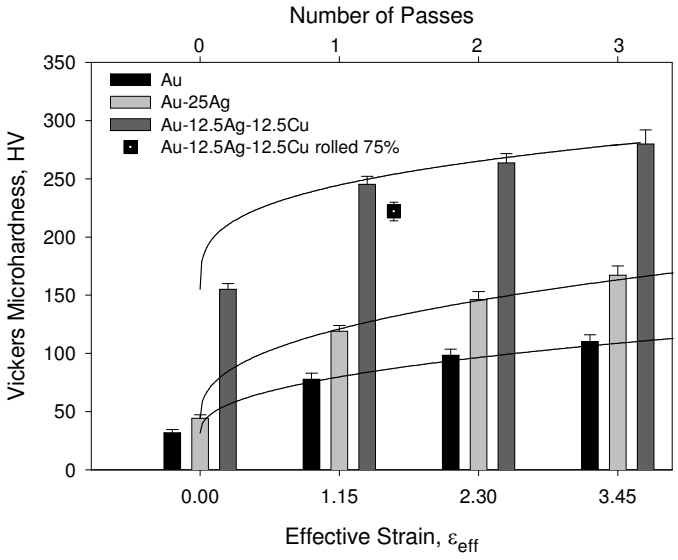

Fig.2. Vickers microhardness as a function of effective strain (number of ECA-passes)

Aging does not alter the strain hardening exponent n, Table 1, i.e. the pre-straining to high strains does not affect the precipitation kinetics appreciably. This is also evident in Fig.3, which shows the hardness dependence on the aging temperature in the ECAP and conventionally rolled AuAgCu alloy. Apparently, the strengthening effect via nucleation of particles is essentially the same in both samples; the aging curves for the rolled and ECAP AuAgCu specimens are nearly parallel to each other. Although it is well-known that cold-working can affect the precipitation kinetics, this effect may tend to saturate after some considerable amount of the imposed strain.

As a matter of discussion it is worth noticing that the ECAP appeared superior in comparison with rolling for strengthening of commercial K18 Au. The hardness of ECAP treated K18 alloy is higher than that of conventionally rolled to 75% area reduction specimen even after the first ECA-pass, as shown in Fig.2. This result is in line with conclusions drawn by Zehetbauer [3], who demonstrated that the equivalent shear stress – shear strain curves of ECAP Cu did not coincide with those of conventional deformation modes, being higher at all strains. He has argued that the benefits of ECAP in comparison with other less-constrained deformation schemes are associated with an enhanced hydrostatic pressure facilitating the uniformity of deformation along the shear plane. Segal [4] has shown that ECAP is beneficial in many aspects when compared to rolling. He emphasized the significance of the deformation mode for structure formation and strengthening, comparing pure and simple shear. It was underlined that the features of simple shear were in early strain localization in the shear bands so that the interaction between them gave rise to rapid nucleation of spatial networks of

high-angle boundaries. In context with this discussion we it might be creditable to notice that the ultimate strength of K18 AuAgCu commercial alloy after ECAP followed by aging exceeds 1 GPa, which has not been achieved in other processing schemes and which is quite attractive for practical utilization in jewelry production. Since the strength of f.c.c. metals typically reaches its maximum after approximately 3-4 passes through the die (see [5], for example) and since the reduction of ductility is not a problem in the present case (in fact, some ductility is even redundant in ordinary Au and Au alloys, which is inconvenient for many artwork), no further ECA-pressing is actually necessary and the desired compromise between the strength and ductility can be readily achieved after first ECA-pressings.

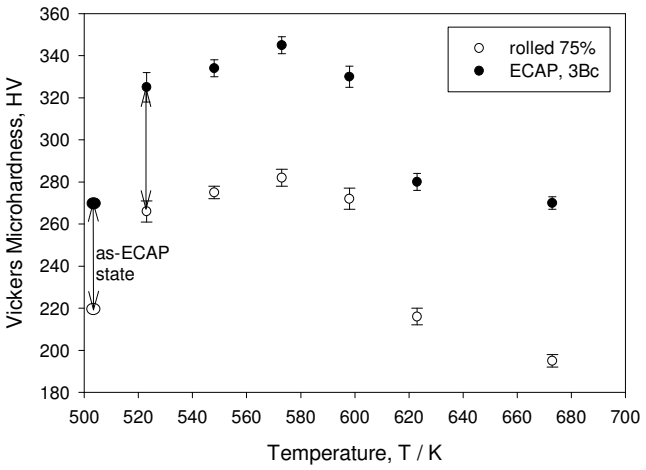

Fig.3. Microhardness of ECAP-manufactured Au-12.5Ag-12.5Cu alloys as a function of aging temperature

Table 1. Strengthening of Au-alloys after SPD

	Au 4N		Au-0.5Ag		Au-25Ag		Au-12.5Ag-12.5Cu				
	O	4Bc	O	Bc	O	Bc	Q	Rolled, 75%		ECAP	
								Rolled	Aged	ECAP	Aged
HV (MPa)	32	96	32	92	44	190	154	222	287	280	345
σ_{UTS} (MPa)	133	245	138	268	185	430	505	685	835	880	1070
n	-	0.42	-	0.42	-	0.30	-	-	-	0.30	0.31

O and Q are fully annealed and quenched state, respectively

Conclusions

Various mechanisms contribute additively to the resultant strength (hardness) of SPD metals at least when the imposed strain is not very large. The strain hardening associated with ECAP at small number of pressings obeys the power low which is typical of dislocation hardening and substructure formation in plastically deforming f.c.c. metals. ECAP is particularly beneficial for strengthening of concentrated solid solutions, probably due to considerable reduction of the stacking fault energy.

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