Structure and Mechanical Properties of CuAu and CuAuPd Ordered Alloys

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The formation of a microstructure during $L1_0$-type ordering in an equiatomic CuAu alloy and ternary Cu-Au-Pd alloys having different initial states has been studied. Results of mechanical tests of the alloys have been given. Methods for improvement of strength properties of the ordered alloys have been discussed.

Introduction

The binary gold-copper system and ternary palladium alloys based on this system are widely used in stomatology, electronics and jewelry. Also, these alloys present interest for basic research since when they are treated at temperatures below some critical point, their structure is rearranged and, as a result, atoms of each species occupy strictly fixed sites in the crystal lattice. This phenomenon is called "atomic ordering" and the resulting crystal lattice is referred to as "a superstructure".

A large body of research showed that the initial face-centered cubic lattice of the equiatomic CuAu alloy is rearranged during atomic ordering to a face-centered tetragonal lattice, in which atoms of each species are arranged in layers parallel to planes of the {001} type. Tetragonality of the crystal lattice is explained by a change of the interplanar spacing "c" between layers of different-species atoms. The degree of tetragonality is determined by the ratio *c/a*. A crystal lattice with this ordered arrangement of atoms is called a $L1_0$ superstructure (Fig. 1). Internal stresses arise in the alloy as the disordered cubic lattice is rearranged to the ordered tetragonal lattice. Therefore, atomic ordering may occasionally lead to spontaneous buckling or cracking of products.

This work attempted to show the evolution of an alloy microstructure, $L1_0$ type, upon ordering. The most interesting results involves the change of microstructure in preliminary cold working alloys. When studying ordering in cold-worked alloys, a combined reaction involving a phase transformation (T) and a recrystallization process (R) is investigated. Depending on the kinetics of these processes,

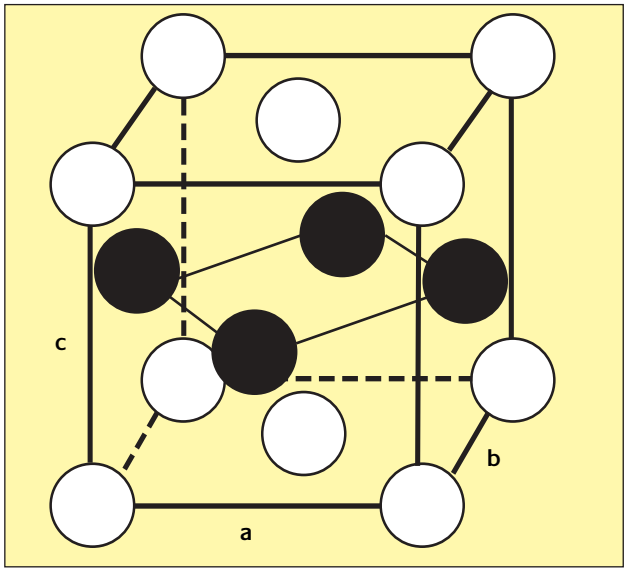

Figure 1
Sketch of a unit cell in the $L1_0$ superstructure. Dark and light spheres denote different species of atoms

both reactions can occur simultaneously or one after the other. For example, ordering is able to precede recrystallization. In this particular case ordering suppresses recrystallization under certain conditions.

When a disordered alloy is subjected to large plastic deformation, a dislocation structure with high dislocation density develops. The rearrangement of such a structure upon annealing substantially depends on the relationship between the rate of ordering and the rates of processes controlling dislocation and redistribution. There may be two limiting cases: "slow ordering", when the dislocations have time to rearrange in the disordered phase; and "fast ordering", when the dislocation structure inherited from the disordered phase turns out to be built into the ordered matrix and forms a kind of a framework. The cause of the last variant results in the change of the translation vectors after disorder-order transformation. A superlattice has translation vectors larger than those of the disordered phase. So the Burgers vectors of the dislocations that form the framework are by no means necessarily the translation vectors of the superlattice.

The difficulties in the rearrangement of the dislocation framework result in the delay of the recrystallization after "fast ordering". This is observed in alloys ordering to the $L1_2$ superstructure, such as Cu_3Au [1] and Ni_3Fe [2]. Note that recrystallization upon ordering after cold working in the CuAu alloy, which orders to a superstructure of the $L1_o$ type, occurs more easily [3] than that in the Cu_3Au alloy. In layered superstructures such as $L1_o$, part of the dislocations inherited from the deformed disordered state retain their mobility. These are dislocations whose Burgers vectors coincide with the shortest translations of the superlattice, i.e. Burgers vectors parallel to {001} planes occupied by like atoms.

Further research into ordered alloys may result in improvement of their consumer properties. The goal of this study was to analyze the structure and properties of an equiatomic CuAu alloy and ternary Cu-Au-Pd alloys ordered after the $L1_0$ type.

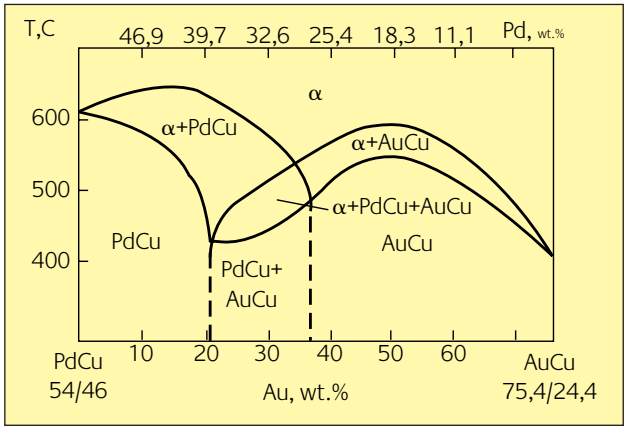

Figure 2
Pseudo-binary phase diagram of the PdCu-AuCu system [4]

Materials and methods

The system of gold-copper-palladium alloys was chosen for the study. Considering the right-hand section of the phase equilibrium diagram (Fig. 2) [4], seven ternary Cu-Au-Pd alloys were smelted. Compositions of the alloys, the degree of the lattice tetragonality, and the width of the two-phase region are given in Table 1. The structure and properties of the alloy 8 were compared with those of a binary equiatomic CuAu alloy (the alloy 1). Their lattices had nearly the same degree of tetragonality [5], but the alloy 1 was free of palladium, while the alloy 8 contained 20 at.% Pd. From Table 1 it is seen that the degree of tetragonality decreased from 0.92 to 0.87 as the palladium concentration increased from 0 to 24.2 at.% in the system. Simultaneously, the critical ordering temperature (T_c) increased considerably from 410°C (CuAu) to 600°C (CuAuPd containing 18.6 at.% Pd) and a two-phase region up to 50° wide appeared (Fig. 2).

A stoichiometric CuAu alloy and ternary CuAuPd alloys were arc-melted in a vacuum (metals of 99.98% purity).

Table 1

No alloy	Compositions of the alloys							Interval of the two-phase region, °C
	Atomic %			Weight %			c/a	
	Au	Pd	Cu	Au	Pd	Cu		
1	50,0	–	50,0	75,6	–	24,4	0,92	Tc=410
2	40,6	7,8	51,6	66,0	6,9	27,1	0,93	500-530
3	37,9	10,0	52,1	63,0	9,0	28,0	0,92	510-545
4	35,3	12,1	52,6	60,0	11,1	28,9	0,91	520-560
5	32,8	14,2	53,0	57,0	13,3	29,7	0,90	540-585
6	27,4	18,6	54,0	50,0	18,3	31,7	0,89	550-600
7	20,6	24,2	55,2	40,0	25,4	34,6	0,87	520-570
8	20,0	20,0	60,0	39,9	21,5	38,6	0,91	515-565

Ingots 6 mm in diameter were homogenized in a 10^{-2} Pa vacuum for 3 h and then were quenched in ice-cold water. The homogenization temperature was 850°C for the ternary alloys and 600°C for CuAu. The alloys were rolled to produce a sheet 0.1 mm thick for examination in a transmission electron microscope (TEM) or deformed by drawing for mechanical testing. The final degree of reduction was 75%. The microstructure of the alloys was analyzed using a JEM-200CX electron microscope. Tensile test samples were 30 mm long and 1.5 mm across.

It should be noted that alloys can be converted to the ordered state using different methods, which are shown schematically in Fig. 3. The first method of ordering applies to alloys, which first are recrystallized in the high-temperature region and then are cooled slowly below the transformation temperature. In what follows, we shall refer to this technique as the "high-temperature" method. The next "low-temperature" method of ordering includes two stages. The alloy is annealed first at a temperature above T_c to form a recrystallized state, which is fixed by quenching. The subsequent thermal treatment of the alloy below T_c converts it to the ordered state. Thermal treatment by the method (III) includes annealing of a pre-deformed alloy below the critical temperature of ordering. Specific features of the formation of the structure and properties of the test alloys, which were observed during the aforementioned methods of ordering, will be discussed below.

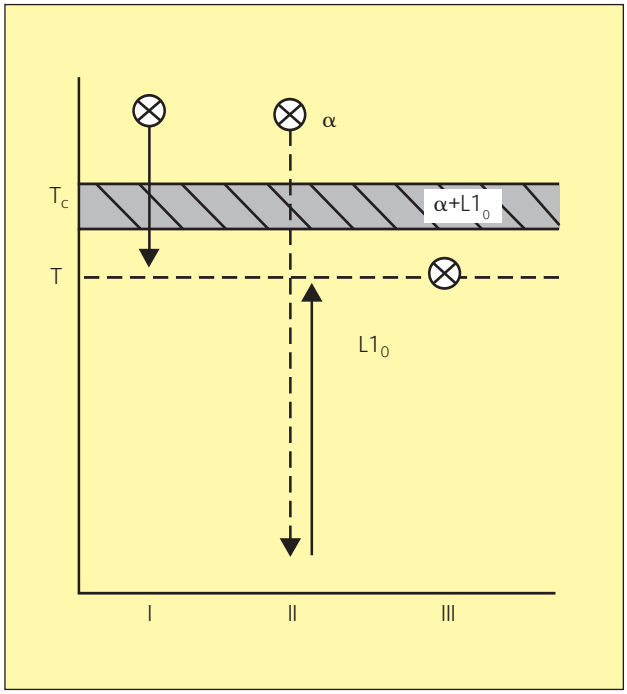

Figure 3
Three different methods of thermal treatment for formation of an ordered state in alloys

Results and discussion

I. Microstructure

The high-temperature method of ordering. It was shown earlier that this method of ordering is optimal for the binary equiatomic CuAu alloy [6]. As the alloy is cooled slowly at temperatures below T_c, it forms a small number of nuclei of a new phase and, consequently, the self-relieve method of ordering is realized. In this case, stresses, which arise around a tetragonal nucleus in the initial cubic matrix, are removed because another nucleus, whose tetragonality *c*-axis is misoriented by 90° relative to the *c*-axis of the first nucleus, appears in the stressed area. Initial stages of the new phase formation in the disordered matrix are clearly seen in Fig. 4a. Stresses at the point of a needle comprising a set of *c*-domains with two orientations are observed. A well-ordered structure represents a set of domain plates joining one another along planes of the {110} type (Fig. 4b). The study of the deformation behavior of this structure revealed that strength properties of the CuAu alloy depend on the domain size (or, which is the same, the number of domain boundaries) [7]. For example, the yield stress of the alloy, which is virtually devoid of domain boundaries, is 115 MPa [8]. If a large number of small domains is present, strength

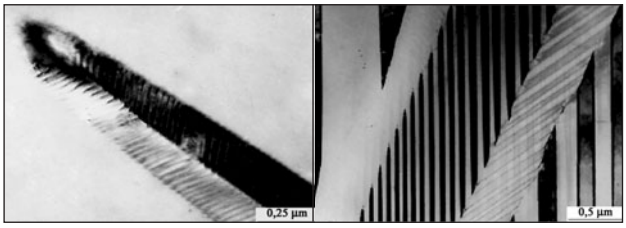

Figure 4a
Formation of a microstructure in an equiatomic CuAu alloy during ordering [6]: a - initial stages of formation of a lamellar structure;

Figure 4b
Well-ordered state

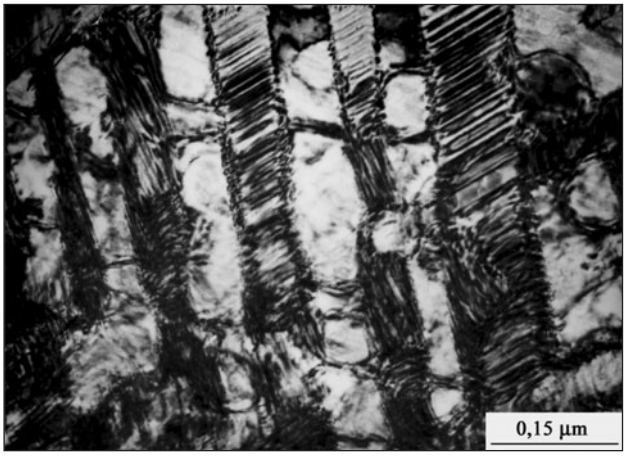

Figure 5
Microstructure of a Cu-Au-Pd alloy (20.0 at.% Pd) after annealing at 600°C for 1 h and cooling to 400°C at a rate of 100 deg./h

Figure 6a
Domain structure of an ordered Cu-Au-Pd alloy (10 at.% Pd) after annealing at 600°C for 1 h: a – fast fall of the temperature at a rate of 100 deg./min

Figure 6b
Slow fall of the temperature at a rate of 100 deg./h

properties of the alloy are improved to $\sigma_{0.2}$ = 750 MPa [9].

Results obtained for CuAuPd alloys, which were ordered by cooling after annealing at a temperature above T_c, are shown in Figs. 5 and 6. It was found that the method of removal of ordering-induced stresses changed with increasing concentration of palladium. A lamellar domain structure was replaced by grains. Each grain represented one *c*-domain. Fine grains could be found nearly in any area of the alloy. In some areas their number was comparable with the volume of the lamellar structure (Fig. 5). Therefore, structures of the binary and ternary alloys had different stability. In the binary CuAu alloy recrystallization nuclei appeared at needle boundaries over the temperature interval from 360 to 400°C and the alloy had to be held at these temperatures for at least 10 days to form the grain structure [6]. The transformation of the lamellar structure to the grain structure took just one hour in the alloy with 20 at.% *Pd*.

Two types of the ordered structure could be produced in alloys with a small (up to 10 at.%) percentage of palladium. A lamellar domain structure was formed in the alloy as the order-disorder two-phase region was passed quickly during cooling (Fig. 6a). Otherwise, the alloy structure proved to be inhomogeneous. Fine-domain areas bordered with coarse domain plates or grains-single domains (Fig. 6b).

Thus, the structure with a large number of domain boundaries could only be formed in CuAuPd alloys containing not more than 10 at.% *Pd*.

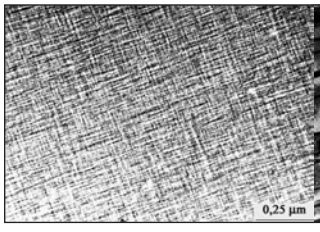

Figure 7a
Variation of the microstructure of an equiatomic CuAu alloy during annealing after quenching from 600°C [6]: a - initial tweed structure

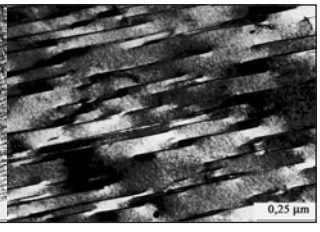

Figure 7b
Coarsening of domains after additional annealing at 370°C for 1 h

The low-temperature method of ordering. This method is not optimal for formation of an ordered structure in the equiatomic CuAu alloy [6]. Quenching of the alloy led to appearance of a large number of nuclei of a new phase (Fig. 7a), which grew rapidly upon subsequent annealing (Fig. 7b). Ordering-induced stresses were not relieved. A strong diffraction contrast in Fig. 7b pointed to a high level of internal stresses, which built up in the alloy during post-quenching ordering. Plasticity of the alloy after this treatment was nearly zero. The material buckled or cracked spontaneously in some cases.

Similarly to the binary copper-gold alloy, a fine-domain structure was formed in the palladium alloys at initial stages of low-temperature ordering (see Fig. 7a). The subsequent stages of the structure formation in the ternary alloys differed fundamentally from the processes observed in the binary equiatomic alloy. Grains of the new phase appeared and increased in size upon further thermal treatment. The alloy did not buckle or crack. Probably, nuclei appeared in strongly stressed areas and removed stresses arising during ordering. A grain structure was formed in the quenched ternary alloys during long-term annealing below T_c (Fig.8). Each grain represented a single *c*-domain.

Ordering after preliminary deformation. The progress of ordering in alloys after preliminary plastic deformation was shown to be very complicated [1]. It was found [10] that the evolution of the microstructure during ordering of pre-deformed alloys should depend considerably on the treatment temperature. The approach [10, 11] was used in this study to analyze ordering processes in a pre-deformed CuAu alloy.

The analysis showed that early stages of the microstructure formation in the CuAu alloy did not differ from those in the undeformed alloy. A random distribution of

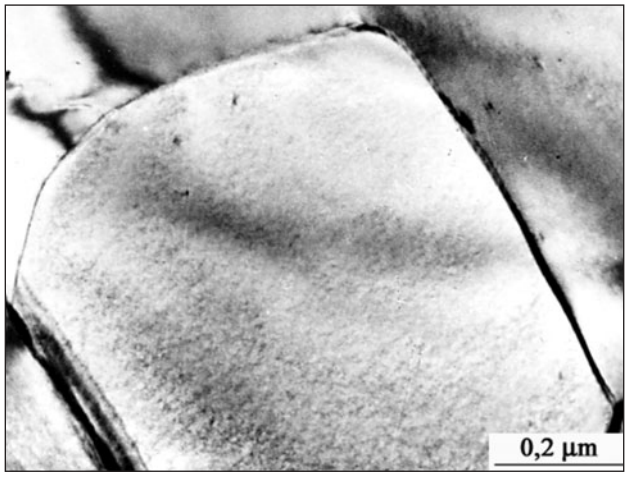

Figure 8
Grain ordered state of Cu-Au-Pd alloy with 18,6 at.% Pd after quenching from 650°C and annealing at 250°C for 5 h

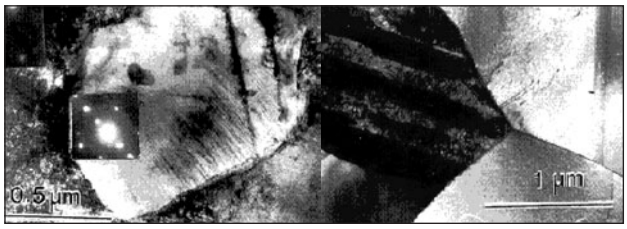

Figure 9a
CuAu cold rolled in a disordered state and annealed at 270°C: a - bright-field image of the microstructure after 1-h annealing. A recrystallized grain is seen in the matrix with a short-range atomic order

Figure 9b
Electron micrograph after 100-h annealing

domains was observed first. The regularity of the microdomain structure was enhanced smoothly and a "tweed" structure (as in Fig. 7a) was formed upon further thermal treatment. The subsequent evolution of the microstructure largely depended on the thermal treatment temperature.

The micrographs in Fig. 9 show the CuAu alloy structure after annealing at 270°C for 1 h (a) and 100 h (b). When the annealing time was short, a typical feature of the microstructure was a recrystallized nucleus in the tweed structure (Fig. 9a). It is interesting to note that new grains were ordered and represented single *c*-domains. An alternative to these single-domain grains was a lamellar structure comprising thin plates of *c*-domains. The alloy, which was annealed at 270°C for 1 h, was ordered, but had a mixed structure including new grains and tweed and lamellar patterns.

If the annealing time at 270°C was increased to 20 h, a considerable fraction of the volume became recrystallized. However, a lamellar structure having no signs of recrystallization and containing relatively large domains about 0.07 µm wide was occasionally preserved. The recrystallized structure occupied a greater volume after 100-h annealing. The average grain size was about 2 µm. However, one could also observe microvolumes with remainders of the lamellar structure, which were squeezed between new grains, and the dislocation density was surprisingly high after such a long annealing time (Fig.9b). Although first new grains appeared very early at 270°C, recrystallization was not complete as long as the lamellar structure was preserved. This behavior was observed over the whole temperature interval below 330°C.

The temperature interval from 330 to 370°C corresponded to the maximum ordering rate in the CuAu alloy [12]. Over this temperature interval a lamellar structure was observed already after a few minutes of annealing (Fig.10a). New grains were not seen in the lamellar structure and a high dislocation density was preserved even after several hours of annealing. A mixed structure was formed at long annealing times. One can see dislocations, polygonal dislocation networks in *c*-domains, subgrains squeezed between *c*-domain boundaries, and new single-domain grains absorbing the lamellar structure while moving. One of these grains extending along a domain boundary is shown in Fig.10b.

At temperatures from 380 to T_c = 410°C the long-period CuAu II phase was stabilized [13]. In accordance with TTT curves, ordering was considerably retarded at temperatures above 380°C [12]. The alloy was ordered after one-hour annealing between 380 and 395°C. Electron diffraction in the selected area along the <100> direction included superlattice spots and well-resolved satellites caused by long-period CuAu II. The volume of the alloy was recrystallized. However, some regions retained the lamellar structure with domains nearly 0.2 µm wide. After 10-h annealing at 395°C a fully recrystallized CuAu II alloy with grains about 10 µm size on the average was obtained. Formation of the microstructure during atomic ordering in the pre-deformed CuAu alloy was described in more detail elsewhere [14].

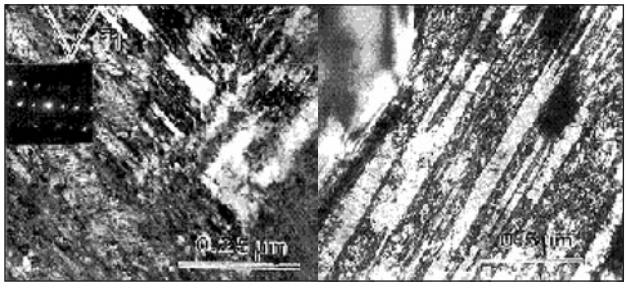

Figure 10a
Microstructure of the CuAu alloy after deformation and annealing at 350°C: a - bright-field image of the microstructure after 1-min annealing

Figure 10b
Formation of a grain in a c-domain structure after 10-h annealing

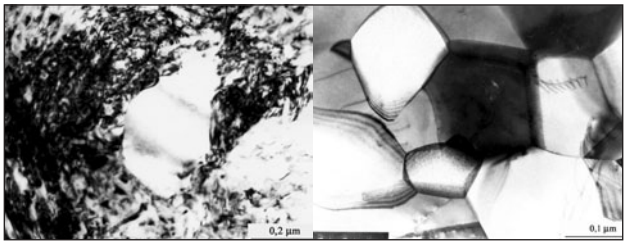

Figure 11a
Annealing of a cold-rolled CuAuPd alloy with 20 at.% Pd at 400°C: a - a new single-domain grain appears in the matrix after annealing for 15 min

Figure 11b
The microstructure after annealing for 2 h

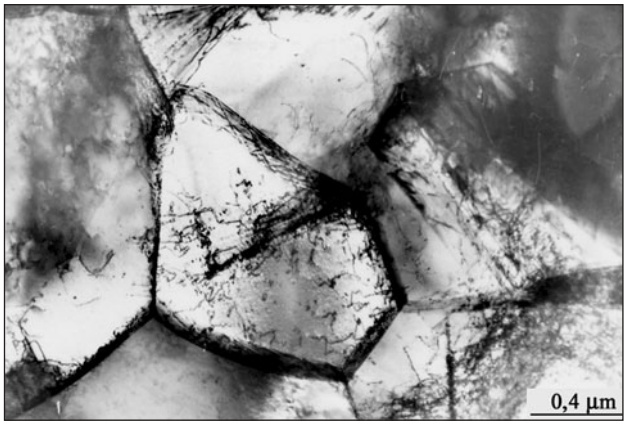

Figure 12
Microstructure of a CuAuPd alloy with 18.6 at.% Pd after cold rolling and annealing at 500°C for 15 min

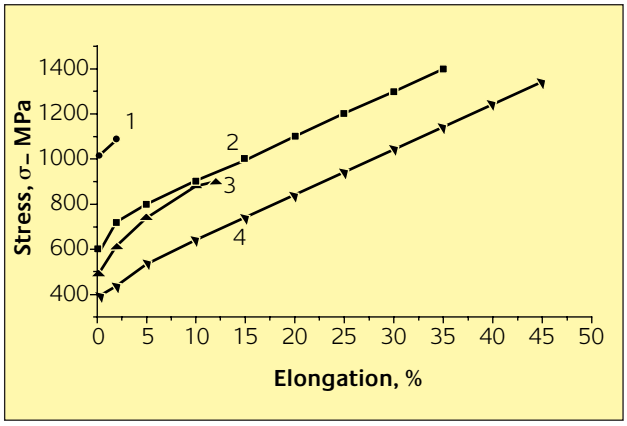

Figure 13
Deformation curves (stress/elongation) for CuAuPd (curves 1, 3 and 4) and CuAu (curve 2) alloys after cold deformation and annealing: 1 - cold deformation by drawing to 75%; 2 - same + annealing at 370°C for 0.5 h; 3 - same as (1) + annealing at 500°C for 1 h; 4 - same as (1) + annealing at 500°C for 40 h

Thus, ordering leads recrystallization over the temperature interval from 330 to 370°C. A temperature anomaly of the recrystallization rate is observed. Indeed, at 270°C recrystallization was complete virtually 100 hours before annealing. At a temperature of 350°C a fully recrystallized state was not obtained after two-month aging. Therefore, the recrystallization rate was faster at the low temperature than at the high temperature of annealing. In line with [10, 11], we explain the retardation of recrystallization at temperatures from 330 to 370°C by an insufficient mobility of dislocations in the ordered matrix, which were inherited after preliminary deformation.

Considering the results described above, ordering-induced changes of the microstructure of pre-deformed CuAuPd alloys were studied over two temperature intervals: in the region of the maximum rate of the long-range atomic ordering (500°C) and at lower temperatures (400°C). The interval near *Tc* was not analyzed since the microstructure was formed over this interval nearly in the same way as at low temperatures.

Figure 11 demonstrates the progress of recrystallization and ordering processes in the pre-deformed matrix of the alloy containing 20 at.% palladium at the annealing temperature equal to 400°C. A nucleus appeared in the fine-domain structure (Fig. 11a). As in the case of the binary CuAu alloy (see Fig. 9a), the electron diffraction pattern of the matrix suggested a short-range atomic order, while the electron diffraction pattern of the formed grain revealed a long-range order. The growing grain was recrystallized since it did not contain a large density of dislocations. The formed grains represented single domains. A distinction from the equiatomic CuAu alloy was a high final rate of the recrystallization process: annealing of the palladium-containing alloys at 400°C for 2 to 5 hours led to formation of a fine-grain structure with grains 0.2 to 0.5 µm in size (Fig. 11b). Let us recall that grain structure was obtained for CuAu after it was annealed for dozens of hours (see Fig.9b).

The microstructure of the alloy containing 18.6 at.% palladium after preliminary cold deformation and holding at 500°C is shown in Fig. 12. The CuAuPd alloys did not form a lamellar structure. The thermal treatment for 15 min. resulted in formation of a grain structure with a high density of dislocations. In some areas one-half of a grain was free of dislocations and their number in the other half was comparable with that in the pre-deformed state. The structure also had grains, which did not contain dislocations at all. The density of dislocations decreased and the grain size increased with the annealing time.

II. Mechanical Properties

Observations in an electron microscope represent a very local method of examination. Therefore, it is difficult to evaluate visually the volume fraction of the material, which was recrystallized. The simplest means in this respect is mechanical tests performed after an appropriate thermal treatment, because the retardation of recrystallization during ordering by the $L1_0$ type should cause formation of high strength properties [15].

Figure 13 presents diagrams of mechanical tests. The curve 1 characterizes mechanical properties of the CuAuPd alloy (18.6 at.% palladium) in the initial state, that is, after plastic deformation to 75%. Mechanical properties of the binary CuAu alloy in the initial 75-% deformed state were nearly equal to those of the ternary alloy and therefore are not shown. The curve 4 corresponds to an ordered recrystallized state of the ternary alloy. The curve 2 in Fig. 13 is given as an example and demonstrates mechanical properties of the CuAu alloy in an ordered non-recrystallized

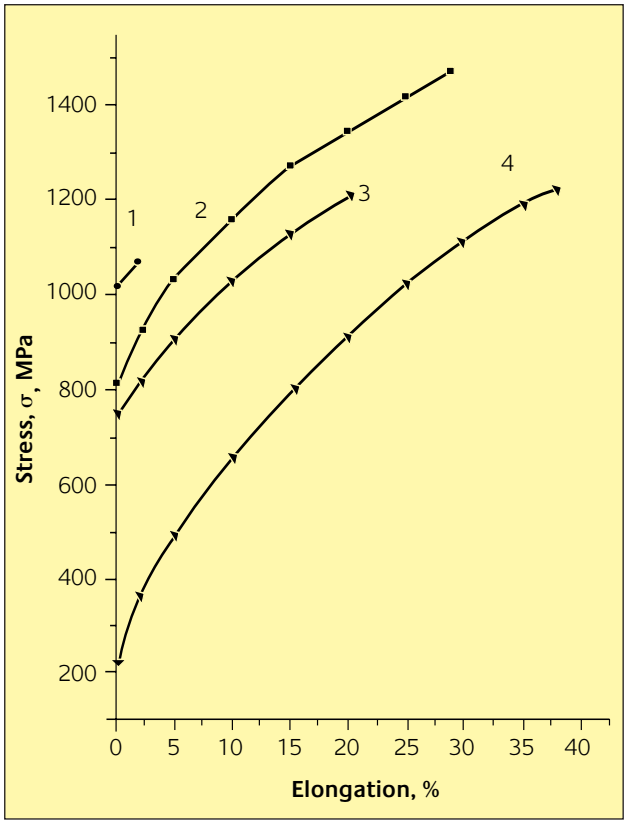

Figure 14

Deformation curves (stress/elongation) for CuAuPd (curves 1 and 2) and CuAu (curves 3 and 4) alloys after cold deformation and annealing: 1 - cold deformation by drawing to 75%; 2 - same + annealing at 400°C for 2 h; 3 - same as (1) + annealing at 450°C for 1 h and slow fall of the temperature at a rate of 100 deg./h; 4 - same as (1) + annealing at 330°C for 25 h

state. The scientific approach, which was used for formation of the structure having an optimal combination of strength and plastic properties, was described in detail elsewhere [15]. This method reduces technically to short annealings over the temperature interval of the maximum ordering rate. The formed structure is similar to the one shown in Fig. 10a. As was mentioned in the foregoing, this treatment leads to quick ordering, while recrystallization is retarded. Dislocations inherited from the initial deformed state had not time to rearrange themselves in the superlattice and formed a kind of a framework. A high density of inherited dislocations provided enhanced strength properties of the alloy. A lamellar structure, which appeared during ordering, acted as a plasticizer.

Short-time thermal treatments of the CuAuPd alloys in the region of the maximum ordering rate did not provide satisfactory strength and plastic characteristics (the curve 3 in Fig. 13). It is seen that the yield stress of the palladium-containing alloy dropped sharply: $\sigma_{0.2}$ values were twice as small as those of the alloy in the initial state. The plasticity of the alloy is small too. The corresponding microstructure is shown in Fig. 12. A high density of dislocations on boundaries and in bodies of the grains probably was not sufficient for considerable strengthening. If the annealing time was increased, the strength properties were impaired more (the curve 4 in Fig. 13).

Thus, short-time annealings, which were performed by analogy with CuAu over the temperature interval of the maximum ordering rate, failed to guarantee an optimal combination of high strength and plastic properties of the pre-deformed alloy with the $L1_0$ superstructure. One may think that recrystallization is not suppressed in CuAuPd alloys.

However, our study showed that an ordered state with single-domain grains 0.2 to 0.5 μm in size (Fig. 11b) was formed in pre-deformed alloys of the CuAuPd system after short annealing at 400°C. Therefore, it is possible to use the known method of grain refinement for improvement of strength properties.

Mechanical tensile properties of the ternary alloy having a fine-grain structure are shown in Fig. 14. The curve 1 characterizes properties of the CuAuPd alloy with 20 at.% Pd in the initial state, i.e. after preliminary deformation to 75%. The curve 2 demonstrates mechanical properties of this alloy after annealing at 400°C for 2 hours. Thus, a fine-grain structure formed in the ternary CuAuPd alloys ensured high strength and plastic properties.

The curve 4 in Fig. 14 shows mechanical properties of an equiatomic CuAu alloy after cold deformation and annealing at 330°C for 25 hours. Subject to this thermal treatment conditions, the gold-copper alloy formed a structure of single-domain grains (see Fig. 9b). Strength properties were very poor, a fact which probably was explained by the size of grains (about 2 μm). A comparison of the microstructures of the binary CuAu alloy and the ternary CuAuPd alloys gave an interesting result. The size of grains formed in the palladium-containing alloys was much smaller at the high temperature. This fact can be explained by a considerable tetragonality of the lattice in the ternary alloys since more *c*-domains of different orientations are required for compensation of elastic stresses that arise during ordering.

The curve 3 in Fig. 14 demonstrates mechanical properties of an equiatomic CuAu alloy, which formed an ordered structure with a maximum possible number of domain boundaries (see Fig. 4b). It is this structure that provided the highest strength properties of the equiatomic CuAu alloy in an ordered state (cf. the curve 2 in Fig. 13). One may see that mechanical properties of the ternary alloy having a fine-grain structure (the curve 2 in Fig.14) were even a little higher.

Conclusions

Considering what has been said above, it may be inferred that a lamellar structure is characteristic, but is not the only possible, of alloys having the $L1_0$-type superstructure.

Processes of the microstructure evolution in CuAuPd alloys end rather quickly in formation of ordered grains.

The study showed that high strength and plastic properties can be formed in alloys with the $L1_0$ superstructure by different methods. The optimal method for a particular alloy can be determined in experiment only. For example, the highest mechanical properties of an equiatomic CuAu alloy can be obtained if its domain structure is refined as much as possible. The retardation of recrystallization and quick ordering provide the best result for other alloys having a lamellar structure (for example, FePd, NiPt, and CoPt [15]). Mechanical properties of CuAuPd alloys, which have not a lamellar structure, are improved thanks to formation of single-domain grains whose size equals tenths of a fraction of a micrometer.

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Dr. A. Yu. Volkov is a Senior Researcher at the laboratory of mechanical properties, Institute of Metal Physics, Ural Branch RAS. He studies phase transformations, the microstructure, mechanical and electrical properties of ordered alloys of gold and noble metals. He is a co-author of 2 patents and 45 scientific papers.

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