IL NUOVO CIMENTO VOL. XLV B, N. 2 11 Ottobre 1966

Vacancy Clusters in Cu_3Au Alloy.

A. CAMANZI and G. SCHIANCHI
Istituto di Fisica dell'Università - Parma

E. RIMINI
Istituto di Fisica dell'Università - Catania
Gruppo Nazionale di Struttura della Materia del CNR

(ricevuto il 29 Settembre 1966)

Rather few electron-microscope observations have been till now performed on the clustering of lattice vacancies in quenched and annealed specimens of noble-metal alloys (1). On the contrary, a great amount of experimental results is available for pure noble metals (2). This note describes the first results of an electron-microscope research on vacancy clustering in Cu_3Au f.c.c. alloy. This alloy shows an order-disorder transition at the critical temperature $T_c \simeq 390$ °C.

Unfaulted dislocation loops, stacking-fault tetrahedra and poligonal faulted dislocation loops have been observed in different areas of the same specimen.

Thin foils of polycrystalline Cu_3Au (*). alloy (purity 99.98%, thickness 50 μm) previously annealed for 50 h at 930 °C in very purified argon atmosphere, were quenched in vacuum (10^{-5} Torr) from a furnace at 900 °C into a silicon oil bath at 0 °C. Some of the quenched specimens were annealed for about 1 h at 100 °C, some others for about 1 h at 200 °C. Following this treatment, the specimens were thinned in a bath of potassium cyanide at 60 °C, and then observed with a Siemens Elmiskope I 100 kV electron microscope with a double-tilt specimen holder (*). Neither traces of vacancy clusters nor detectable nuclei of order have been observed in quenched and unannealed specimens (thinned in a 70% acetic acid and 30% perchloric acid bath at 0 °C).

The observed defects ranged widely in shape and size both from one to another specimen, and from one to another area of the same specimen. Areas of specimens quenched from 900 °C and annealed for 1 h at 100 °C are shown in the micrographs of Fig. 1 and 2. Stacking-fault tetrahedra ($\sim 3 \cdot 10^{13}/cm^3$) and poligonal faulted dislocation loops,

(1) G. THOMAS and J. WASHBURN: *Rev. Mod. Phys.*, **35**, 922 (1963).

(2) R. M. J. COTTERIL: *Lattice Defects in Quenched Metals* (New York, 1965), p. 97.

(*) Supplied by Metalli Preziosi.

(*) Electron-microscope observations have been carried out at the Laboratorio di Microscopia Elettronica, Istituto di Fisica dell'Università, Bologna.

in rather low concentration ($< 10^{13}/cm^3$), are visible. Figures 3 and 4 show micro-

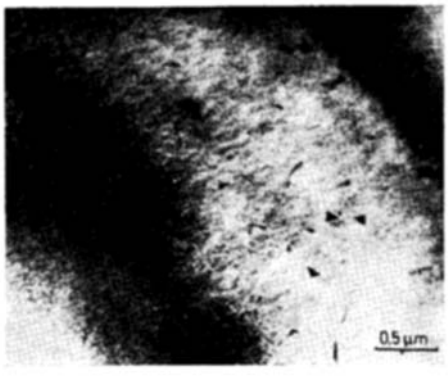

Fig. 1. – Stacking-fault tetrahedra in a specimen of Cu_3Au quenched from 900 °C into a silicon oil bath at 0 °C, and annealed for 1 h at 100°C The electron-beam direction is close to [1 2 3].

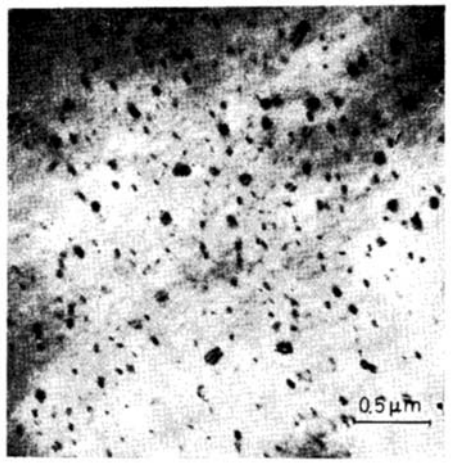

Fig. 3. – Unfaulted dislocation loops in a specimen of Cu_3Au quenched from 900 °C into a silicon oil bath at 0 °C and annealed for 1 h at 200 °C. The electron-beam direction is close to [1 1 0].

graphs of areas of specimens quenched from 900 °C and annealed for 1 h at 200 °C. Unfaulted dislocation loops ($\sim 10^{14}/cm^3$), of the kind mostly observed in quenched Al [3] are evident in Fig. 3.

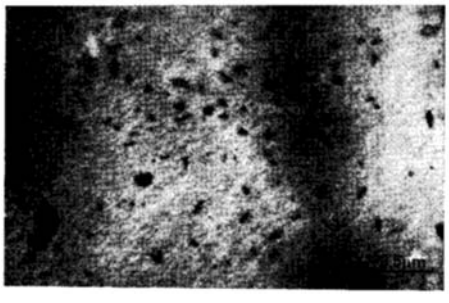

Fig. 2. – Stacking-fault tetrahedra and faulted dislocation loops in a specimen of Cu_3Au quenched from 900 °C into a silicon oil bath at 0 °C, and annealed for 1 h at 100 °C. The electron-beam direction is close to [1 2 3].

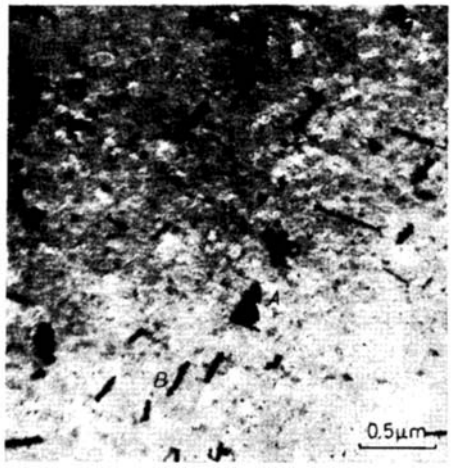

Fig. 4. – Stacking-fault tetrahedra in a specimen of Cu_3Au quenched from 900 °C into a silicon oil bath at 0 °C, and annealed for 1 h at 200 °C. The electron-beam direction is close to [1 1 0]. For the explanation of *A* and *B* see the text.

Triangular shape *A* (max. linear dimension 3000 Å, density $\sim 10^{13}/cm^3$) and linear shape *B* (max. linear dimensions 3000 Å, density $\sim 10^{13}/cm^3$) defects are well visible in the micrograph of Fig. 4. The linear defects *B* are probably stacking-fault tetrahedra truncated during the electrothinning of the specimen.

(3) P. B. HIRSCH, J. SILCOX, R. S. SMALLMAN and K. H. WESTMACOTT: *Phil. Mag.*, **3**, 897 (1958).

Stacking-fault tetrahedra of rather smaller size than those of Fig. 4, and similar in dimension to those observed in pure gold, quenched and annealed at the same temperature [4], are evident in some other micrographs. The maximum density and the maximum linear dimension of the defects, and the corresponding vacancy concentration Cv_{max} are given in the Table.

Defects	Density (N/cm^3)	Max. linear dimension (Å)	Cv_{max}	T (°C)
Dislocation loops	10^{14}	1000	$3 \cdot 10^{-4}$	200
Stacking-fault tetrahedra	$2 \cdot 10^{13}$	3000	10^{-4}	200
Stacking-fault tetrahedra	$3 \cdot 10^{13}$	2500	10^{-4}	100

The vacancy concentration is in agreement with recent experimental results [5].

The electron-diffraction patterns of some quenched and annealed specimens show superlattice reflection spots, probably due to the presence of small ordered nuclei formed during the annealing of quenched vacancies. The diffraction contrast image of the nuclei of order is not evident in the published micrographs, although their presence is supported by the first dark-field micrographs, now available.

The presence of tetrahedra and of poligonal faulted loops agrees with the results of a recent research on zone-refined Cu [6], in spite of the different purity of the adopted material, which seems to be critical in pure metals [1]. Similar defects have been observed also in pure gold [7]. In both cases the presence of unfaulted dislocation loops, typical of Al, is not evident.

The presence of unfaulted prismatic dislocation loops from one side and of stacking-fault tetrahedra and faulted poligonal dislocations loops from the other, in different areas of the same specimen, perhaps might be correlated both with the purity of the adopted material and with a variation of stacking-fault energy, following the different amount of order in the different areas. Recent works [8-9] pointed out that the stacking-fault energy depends on the amount of order, decreasing with the increase of the latter.

Further systematic observations with materials of various purity, now in progress, would clarify the above situation.

* * *

One of us (E.R.) is indebted to the Ministero della Pubblica Istruzione for a grant.

(4) J. SILCOX and P. B. HIRSCH: *Phil. Mag.*, **4**, 72 (1959).

(5) S. BENCI, G. GASPARRINI, E. GERMAGNOLI and G. SCHIANCHI: *Journ. Phys. Chem. Solids*, **26**, 2059 (1965).

(6) L. M. CLAREBROUGH, R. L. SEGALL and M. H. LORETTO: *Phil. Mag.*, **13**, 1285 (1966).

(7) L. M. CLAREBROUGH, R. L. SEGALL, M. H. LORETTO and M. E. HARGREAVES: *Phil. Mag.*, **9**, 377 (1964).

(8) M. J. MARCINKOWSKI and L. SWELL: *Acta Met.* **11**, 373 (1963).

(9) M. J. MARCINKOWSKI: in *Electron Microscopy and Strength of Crystals* (New York, 1963), p. 333.