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FORMATION OF AN INTERMEDIATE PHASE IN ALUMINIUM-GERMANIUM SYSTEM

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INTRODUCTION

THE interesting potentialities of the Duwez technique of rapidly quenching metals and alloys from the liquid state (also known as splat cooling or splat quenching) are now well recognized.[1,2] In addition to striking extension of terminal solid solubility limits in various binary systems, formation of several amorphous and new intermediate phases has been achieved, thanks to the spectacular cooling rates (10^5–10^8 deg. C/sec.) and consequent drastic undercooling associated with this technique.

We report here on the crystal structure of an aluminium-germanium (Al-Ge) phase, produced by application of the Duwez technique. Under equilibrium conditions the Al-Ge system exhibits a eutectic at 30·3 at.% Ge and 424° C. Al is insoluble in Ge in the solid state, but dissolves a maximum of 2·8 at.% Ge at the eutectic temperature. Predecki *et al.*[3] reported extension of solid solubility of Ge in Al on splat cooling, without any indication of the actual increase in solubility. The formation of a complex and unidentified intermediate phase around the eutectic composition was also briefly reported by them. The new phase identified by us in the present work was also obtained in an Al-30 at.% Ge alloy.

EXPERIMENTAL PROCEDURE

Weighed quantities of Al and Ge (both 99·99% pure) were melted in an evacuated fused silica capsule. The alloy was then homogenized for 15 hours at a temperature of 400° C.

The technique of liquid quenching has been reported earlier.[4] In the present work it mainly consisted of loading 50-100 mg. of the alloy in a resistance-heated graphite crucible with a nozzle at the bottom and heating to a temperature about 50° C. higher than the liquidus. The molten globule of the alloy was then expelled by means of a shock wave on to a copper substrate at room temperature. The splat-cooled products were thin foils of variable thickness (upto 15 μm) and could be easily peeled from the substrate. They were examined in a Philips 114·6 mm. dia. camera with nickel-filtered Cu K_a radiation (λCu K_a = 1·54051 Å).

EXPERIMENTAL RESULTS

The Debye-Scherrer patterns consisted of weak Al reflections and no less than 35 other lines, none of which corresponded to Ge. Many of the extra lines were diffuse, the doublets being unresolved in case of high-angle reflections. All the reflections could, however, be satisfactorily indexed on the basis of a new phase with a tetragonal structure and with lattice parameters $a = 13 \cdot 03$ Å, $c = 12 \cdot 04$ Å and $c/a = 0 \cdot 924$ Å. Table I gives the relative intensities of the first 30 extra lines and the observed and calculated values of $\sin^2\theta$.

Deformation at room temperature did not transform the new phase, but only broadened the X-ray reflections further. All the lines due to the extra phase disappeared and gave way to the Al and Ge reflections after annealing at 300° C. for 30 minutes.

DISCUSSION OF RESULTS

The present study has shown that a new intermediate phase in the Al-Ge system can be produced by quenching the molten eutectic alloy to room temperature. The earlier investigators[3] reported the formation of a new phase only when the substrate was maintained at liquid nitrogen temperature,

TABLE I

Observed and calculated $\sin^2\theta$ values of the metastable phase in aluminium-30 at.% germanium alloy

Structure: TETRAGONAL
Lattice parameter: $a = 13 \cdot 03$ Å $\simeq \sqrt{10}\, a_{FCC}$
$c = 12 \cdot 04$ Å $\simeq 3\, a_{FCC}$
$c/a = 0 \cdot 924$

hkl	$\sin^2\theta_{calc.}$	$\sin^2\theta_{obs.}$	$I_{obs.}$
220	0·0280	0·0296	vw
030	0·0315	0·0317	vw
113	0·0439	0·0442	ms
230	0·0455	0·0462	vvw
032	0·0475	0·0479	vvw
023	0·0509	0·0518	vvw
040	0·0560	0·0557	vw
330	0·0630	0·0632	vw
004	0·0656	0·0658	w
240	0·0700	0·0700	vw
133	0·0719	0·0718	w
042	0·0824	0·0819	w
242	0·0864	0·0858	s
152	0·1074	0·1075	vw
161	0·1336	0·1322	w
006	0·1476	0·1487	vw
116	0·1546	0·1549	ms
045	0·1585	0·1598	ms
163	0·1664	0·1688	vw
245	0·1725	0·1728	vw
271	0·1896	0·1890	vvw
236	0·1931	0·1925	vvw
336	0·2116	0·2121	vvw
246	0·2176	0·2168	w
047	0·2569	0·2576	vvw
090	0·2835	0·2835	vvw
257	0·3024	0·3014	vvw
048	0·3184	0·3176	vvw
067	0·3271	0·3274	vw
00·10	0·4100	0·4115	vvw

s = strong; ms = medium strong; w = weak; vw = very weak; vvw = very very weak.

The new metastable phase has been shown to be based on a rather large tetragonal unit cell. Such large unit cells are not uncommon in splat-cooled products. In the Au-21 at.% Ge alloy, a large tetragonal unit cell containing 176 atoms per unit cell[5] and in Au-25-50 at.% Si alloys an f.c.c. unit cell containing 500 atoms per unit cell[6] have been reported earlier.

The tetragonal unit cell of the new Al-Ge phase can be conceived of as forming from 30 unit cells of f.c.c. Al as shown in Fig. 1. The face diagonal of the rectangle formed by stacking three f.c.c. unit cells may be visualized as forming the 'a' parameter of the tetragonal unit cell (*i.e.*, $a_{Tet} = \sqrt{10}\, a_{FCC}$). The '$c$' parameter corresponds to almost exactly 3 times the lattice parameter of the f.c.c. unit cell ($a_{Al} = 4 \cdot 049$ Å). The suggestion that

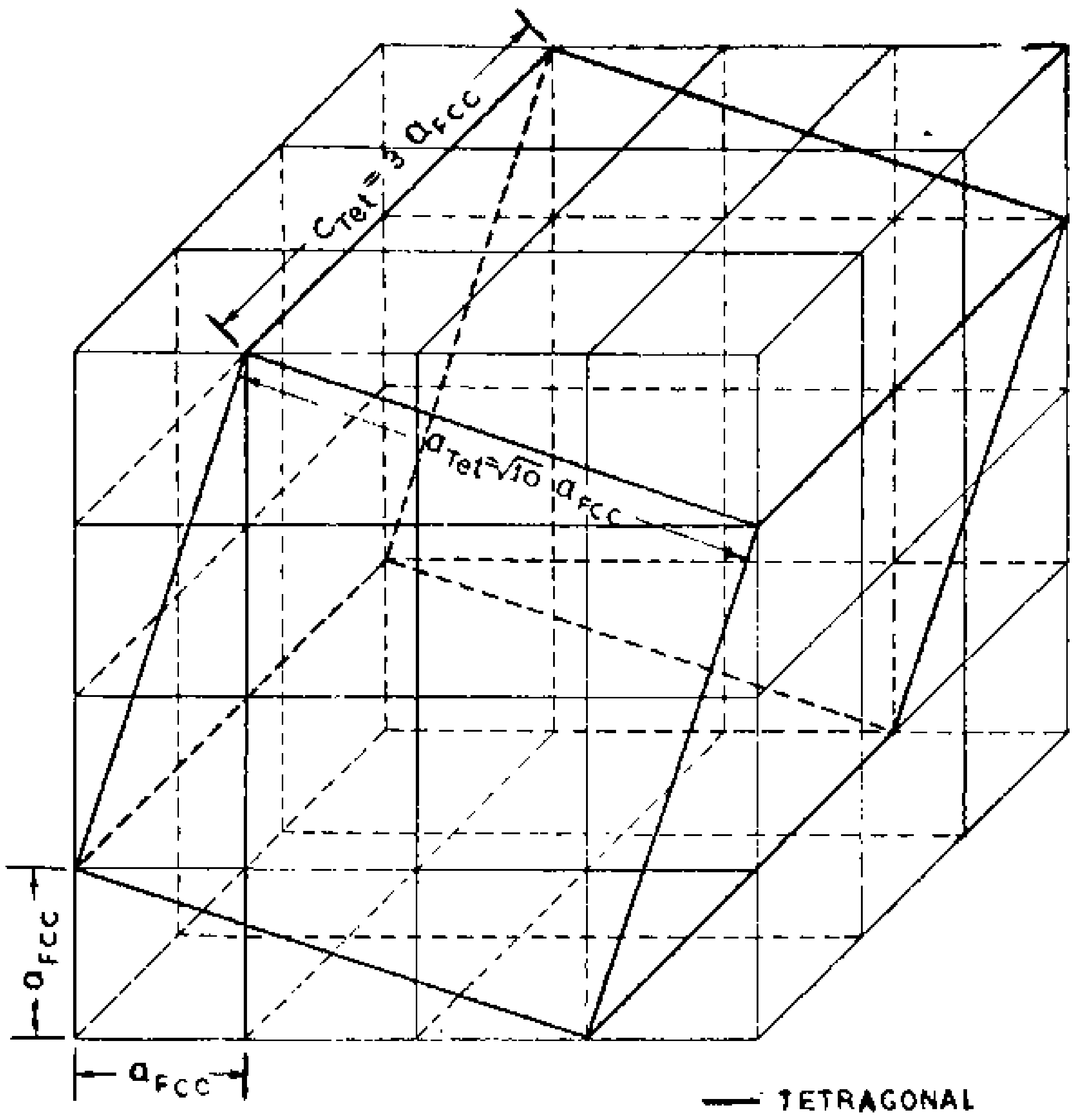

FIG. 1. Formation of the large tetragonal unit cell of the new Al-Ge phase with $a_{Tet} \simeq \sqrt{10}\,a_{FCC}$ and $c_{Tet} \simeq 3\,a_{FCC}$ from 30 unit cells of Al.

the tetragonal unit cell is formed by the stacking of three f.c.c. unit cells in all the three directions receives some support from the frequent occurrence of the number 3 or its multiples in indices (*hkl*) of the X-ray reflections from the new phase (Table I). Assuming 120 atoms per unit cell on the basis of the same close packing as in the f.c.c. structure the atomic volume works out to 17·03 Å^3 from the dimensions of the tetragonal cell. This value is almost identical with the extrapolated atomic volume (17·02 Å^3) for the Al-30 at.% Ge alloy[7] and further strengthens the suggested model for the unit cell of the new phase.

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SOME PRESSURE OSCILLATIONS OBSERVED IN INDIA AND THEIR PROBABLE ASSOCIATION WITH THE CHINESE NUCLEAR TEST 1965

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INTRODUCTION

WELL-MARKED oscillations extending for more than four hours were observed in the microbarograph records on 15th May, 1965 at Okha (22° 29′ N. 69° 07′ E.) in Gujarat State. Some stations north of latitude 18° N. showed abrupt changes in the barograph traces as late as on 20th May, 1965. These suggested the possibility of propagation of pressure waves in the Indian latitudes as a result of the Chinese nuclear test during this period and led to detailed examination of the records of many stations in the neighbourhood. Figure 1 shows the stations whose barograph records were examined and which recorded significant impulses.

'ROUND-THE-WORLD' WAVES

Authentic reports about the exact time of explosion are not available. According to a special report[1] by Edward Neilan, the second blast was detonated at Lop Nor on 13th May, 1965, the actual time of explosion being not mentioned. Pressure waves can exist for long period without absorption and therefore it is not unlikely that the above pressure fluctuations recorded in India resulted from this explosion.

After the Russian nuclear tests at Novaya Zemlya in October, 1961, special observations at the Atomic Weapons Research Establishment, Essex,[2] with high sensitivity barographs showed clear signals of large amplitude 76 hours after the explosion. This was attributed to the waves being successively reflected at the antipodes with relatively little absorption or scattering. Similar studies at Sodankyla, Finland[3] after the same explosion showed microbarograph deflection 38 hours later and these were interpreted as due to "round-the-world waves" both in the forward and backward direction with mean velocity of 311 metres/sec. It was therefore considered worthwhile to examine the traces in the present case in order to see if the fluctuations were due to direct or 'round-the-world' waves after the Chinese explosion.