SovietRxiv

Soviet-era science, translated into English

CRYSTALLOGRAPHY

1963

SovietRxiv

View the original and related papers at https://sovietrxiv.org/items/ru-196301.29671

Source: Math-Net.Ru and CyberLeninka. Machine translation. Verify with the original.

SovietRxiv

Fig. 1

Figure 1: Fig. 1

Abstract

Full Text

CRYSTALLOGRAPHY

A. V. SANDULOVA

PREPARATION AND EVALUATION OF SOME PROPERTIES OF FILAMENTOUS AND NEEDLE-LIKE SINGLE CRYSTALS OF SOLID SOLUTIONS OF THE Ge–Si SYSTEM

(Presented by Academician N. V. Belov, 20 VIII 1962)

By the method described in ([1]), filamentous (diameter up to 20 μ) and needle-like (diameter from 20 to 800 μ) single crystals of solid solutions of the Ge–Si system of different compositions were obtained.

The single crystals were grown in a closed quartz ampoule divided by a constriction into two unequal parts: a dissolution zone and a crystallization zone. In the first, more capacious part of the ampoule were placed weighed portions of single-crystalline germanium and silicon corresponding to a definite composition of the solid solution, and a weighed portion of the solvent component, for which bromine was used. An attempt to use for this purpose certain other substances (sulfur, selenium, tellurium) did not give positive results. The bromine was introduced into the ampoule in the solid state.

Fig. 1

The charged ampoule was immersed in liquid nitrogen, connected to a vacuum system, evacuated to a pressure of 10^{-5} mm Hg, and sealed under vacuum. The sealed ampoule was placed in a furnace so that the temperature of the end of the ampoule in which the weighed portions were located was 1150–1250°; the temperature of the end of the ampoule beyond the constriction was 800–1000°. Lower temperatures led to a slowing of the process and therefore were used less often. The vapor pressure of the solvent in all experiments did not exceed 4 atm. The germanium and silicon vapors formed as a result of dissolution of the corresponding crystals were transported beyond the constriction into the colder part of the ampoule, where their condensation led to the nucleation and growth of mixed crystals.

When the cooled ampoules were opened, at their "cold" end there was found

SovietRxiv

Fig. 2

Figure 2: Fig. 2

Fig. 3

Figure 3: Fig. 3

a polycrystalline layer adjacent to the wall of the ampoule—a cluster of many small single crystals, on which, as on a substrate, threads and needles nucleated and grew from the apex, forming bundles (Fig. 1). The centers from which the growth of the threads and needles proceeded were also individual single crystals that had grown on the walls of the ampoule (Fig. 2).

The whisker crystals obtained by us have, along their entire length, approximately the same cross section and have no bends caused by a change in the direction of growth. The maximum length of the whiskers reached 30 mm. The growth of needle-shaped crystals was sometimes accompanied by their branching and by a change in the initial direction. To investigate the single-crystal nature of such crystals at the place of their bend, Laue photographs were taken; these showed the absence of twinning. The crystals have a clean and even surface that requires no additional mechanical treatment.

We took epigrams from a plane perpendicular to the direction along which various needle-shaped crystals were elongated. In all cases this was the (111) plane, and crystal growth occurred along the ⟨111⟩ direction. The simple forms of the crystals were established by measuring the needle-shaped crystals on a two-circle goniometer of the GD-1 type. All measured needle-shaped crystals are a combination of the following simple forms: cube {100}, octahedron {111}, tetragon-trioctahedron {112}, and rhombododecahedron {110}.

Fig. 2

The study of whisker crystals showed that, when their thickness is small—on the order of several microns or less—facets do not appear on them. Their cross section is round.

Fig. 3

X-ray structural analysis of many of the obtained specimens of whisker and needle-shaped crystals of solid solutions of germanium and silicon confirmed their single-crystal nature and homogeneity. The specified compositions of the solid solutions were in many cases refined by determining the lattice parameter of the obtained crystals and finding the composition corresponding to this value of the parameter on the straight line connecting the lattice parameters of pure germanium and silicon. The discrepancies between the specified and the found compositions were substantial and considerably exceeded those for the case of growing bulk single crystals at a small temperature gradient.

SovietRxiv

Favorable conditions for the nucleation and growth of whisker and needle-

single crystals during crystallization from the gas phase under the conditions of our experiments are created as a result of the action of unsaturated vapors of the solvent component on the crystals and vapors of the starting substances. Unsaturated bromine vapors dissolved germanium and silicon at a rate significantly exceeding the crystallization rate on the parent material, and promoted the transfer of substances converted into vapor from the hotter to the colder part of the ampoule. In the colder part of the ampoule, under the influence of the solvent vapors, there apparently arose, on the whole, an insignificant supersaturation of the gas phase with respect to the dissolved substances, which favored the nucleation and growth of crystals predominantly from a single screw dislocation.

Table 1

Batch	d, μ	$\sigma_{\max}$, kg/mm^2	$\varepsilon_{\max}$, %	$E \cdot 10^{-4}$, kg/mm^2
10 at.%				
Si				
84/3	17	360	0.68	5.3
84/3	20	217	0.87	2.5
84/3	25	133	0.83	1.6
84/3	50	83	0.26	3.2
84/3	90	45	0.15	3
84/3	130	40	0.1	4
84/3	140	34	0.1	3.4
55 at.%				
Si				
96/34	8	752.0	1.6	4.7
96/34	13	416.0	2.6	1.35
96/34	15	375.0	1.5	2.5
96/34	20	114.4	2.86	4
96/34	90	75.3	0.21	3.3
96/34	130	69.6	0.54	1.4

We have estimated the mechanical properties of the grown filamentary and acicular crystals using the bending-deformation method. Deformations and stresses in the elastic region for calculating specimens with diameters from 1 to 30 μ were computed from the expression: $E = 4Fl^3/3\pi d^4\delta$, where l is the length of the specimen; d is the diameter; δ is the deflection; $\varepsilon_{\max}$ is the maximum deformation; $\varepsilon_{\max} = \dfrac{d}{D} \cdot 100$; D is the diameter of the twisting loop. For thicker specimens the formulas used were:

SovietRxiv

$$\varepsilon_{\max} = \frac{l}{l^2/4\delta + \delta} \cdot 100; \qquad \sigma_{\max} = E\varepsilon_{\max}.$$

Table 1 gives some data characterizing the mechanical properties of filamentary and acicular crystals of solid solutions of germanium and silicon. For the measurement of mechanical properties, individual filamentary and acicular crystals obtained in different experiments were selected. The tests were carried out at room temperature.

From Table 1 it follows that filamentary and acicular single crystals possess high mechanical strength and elasticity. To illustrate the elasticity, Fig. 3 shows a microphotograph of a filamentary single crystal twisted for determination of the mechanical deformation. The values of maximum stresses for filamentary crystals of the solid solution containing 55 at.% Si obtained by us substantially exceed the largest known stress value for silicon filamentary crystals (d = 16–28 μ), equal to 500 kg/mm^2 ([2]).

Table 2

Si in solid solution, at.%	Specific resistance of starting material, $\Omega \cdot$ cm	Specific resistance of starting material, $\Omega \cdot$ cm	Specific resistance of grown crystals, $\Omega \cdot$ cm
	Si	Ge	
10	27	27.5	200–700
55	27	27.5	3500–6150

In addition to the study of mechanical strength, we systematically measured the specific resistance of various specimens of filamentary and acicular single crystals of solid solutions. Some data are given in Table 2. The significant increase in the specific resistance of the grown crystals indicates that crystallization of filamentary and acicular crystals by our method is accompanied by an increase in their purity in comparison with the starting material.

Received
3 VIII 1962

CITED LITERATURE

1. A. V. Sandulova, P. S. Bogoyavlenskii, M. I. Dronyuk, DAN, **43**, No. 3 (1962).

2. G. Z. Pearson, W. T. Read, W. Z. Feldman, Acta Met., **181** (1957).

Note: Figure translations are in progress. See original paper for figures.

SovietRxiv

Source: Math-Net.Ru and CyberLeninka. Machine translation. Verify with the original.