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A Method for Preparing Sphere-Shaped Single Crystals of High Melting Metals Without Crucible

For the investigation of different directed crystal properties sphere-shaped single crystals have proved to be useful. Crucible methods are not qualified for the manufacture of monocrystals of high melting metals or their alloys. Sphere-shaped monocrystals can be turned off from cylindrical ones (PAWEL, CATHCART, CAMPBELL). Apart from the fact that this method fails, e.g. for the very hard metals molybdenum and tungsten, an additional chemical treatment and a subsequent vacuum tempering are required, if an originally uncontaminated surface of a crystal with high quality is wanted (STRINGER).

A method is developed enabling sphere-shaped single crystals to be produced without crucible and subsequent treatment: Wire-or rod-shaped basic material is fixed vertically on one end in a high vacuum chamber (residual gas pressure lower than $5 \cdot 10^{-6}$ mm Hg, if possible) in such a manner that the free lower end can be heated up to the melting temperature by means of electron impact (ring-shaped tungsten cathode; specimen as anode), whereby a drop ist formed hanging on the specimen. The heating power being switched off, the drop solidifies sphere-shaped and monocrystalline.

As an example Figure 1 shows the structure of a tantalum specimen in a micrograph.

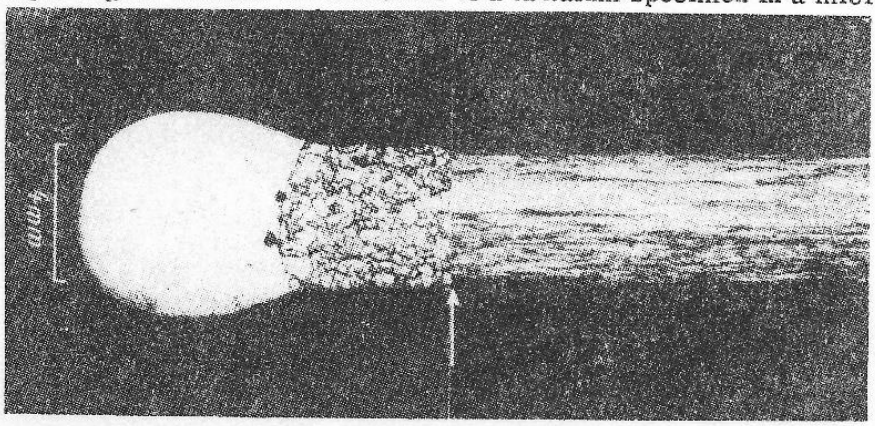

Fig. 1. A monocrystalline sphere of tantalum on a rod-shaped specimen after polishing and etching (vertically shortened by micrograph montage)

Below a smooth, electrolytically polished surface of the monocrystalline sphere with the adjacent rod region is to be seen, electrolytically etched and thus rendering visible the recrystallization structure. Above the fibre structure of the cold-drawn basic material 4 mm in diameter is shown. Etching the monocrystalline surface an etch pit density of $6 \cdot 10^6$ cm^{-2} was obtained. - Occasionally spheres are formed containing two or more crystallites.

Sphere-shaped single crystals were also prepared from tungsten, niobium and zirconium specimens of different size. Likewise the method is suitable for alloys.

From a theoretical estimate of the heat balance it may be concluded that the crystallization front is moved from the interface rod/sphere to the vertex of the latter. Figure 1, however, shows several small shrinkage cavities as black spots at the interface rod/sphere, the formation of which is merely to be ascribed to an opposite movement of the crystallization front. - The clarification of this question essential to the understanding of the crystal growth mechanism in this case would necessitate further investigations.

REFERENCES

PAWEL, R.E., CATHCART, G.V., CAMPBELL, J.J.: Acta Met. 10, 149 (1962)
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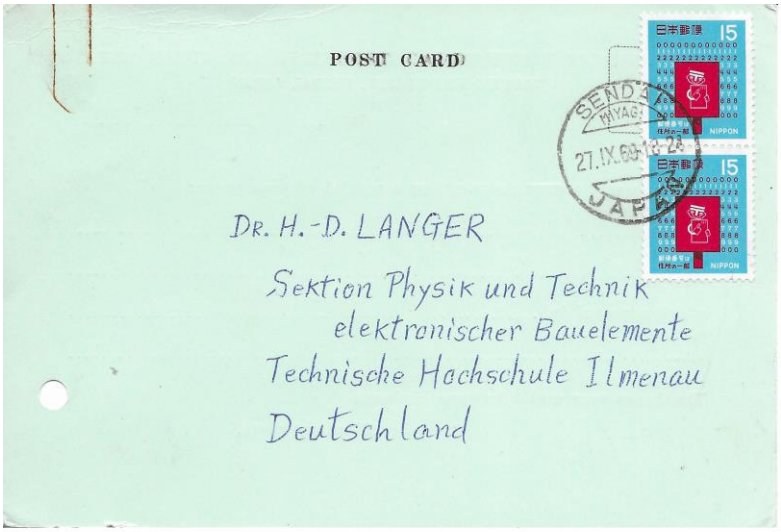
Anmerkung
Es gab zu dieser Veröffentlichung mehrfach Sonderdruckanforderungen durch den Eisernen Vorhang, was einen jungen DDR-Physiker selbstverständlich mit Stolz erfüllte und zugleich herausforderte. Hier ein Beispiel:



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Date Sept. 22, 1969

Dear Sir:

I would appreciate receiving a reprint of your paper:
A Method for Preparing Sphere-Shaped Single Crystals of High Melting Metals
which appeared in Kristall und Technik 1 (1969) K 5.

Thank you very much for your kindness.

Sincerely yours,
Hiroshi IWASAKI