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Some New Whiskers

Filamentary crystals have been known for many years (1), but the recent discovery of spontaneous filamentary growths of several metals by Compton, Mendizza, and Arnold (2), and the subsequent observation by Herring and Galt (3) that these filamentary growths are many times stronger than massive crystals, have resulted in intensified interest in their growth and properties. Several methods of growing "whiskers" (as these crystals are now called) have been developed. Growth by condensation from vapor (4), electrolytic deposition from solution (5), and chemical reactions (6) have been reported. During the course of a current study of the growth kinetics and physical properties of whiskers, whiskers of palladium, β -manganese, and an intermetallic compound, manganese silicide (Mn_5Si_3) , have been obtained. Whiskers of these three materials have not been reported previously.

The palladium whiskers are produced by the thermal decomposition of liquid palladium dichloride at 960°C and are between 1 and 10 mm in length. The β-manganese whiskers, about 0.25 mm long, are grown by the hydrogen reduction of liquid manganous chloride at 940°C in alumina reaction vessels. The salt was distilled prior to reduction to remove moisture and other impurities. The manganese silicide whiskers are formed during the hydrogen reduction of the manganous chloride in the presence of silicon dioxide at 940°C and are also about 0.25 mm long. All these 16 AUGUST 1957

whiskers are in the neighborhood of 2 μ in diameter. The reactions are carried out in gastight refractory tubes through which argon or hydrogen flows at a linear velocity on the order of 1 cm/sec.

Whiskers produced by the thermal decomposition of palladium dichloride nucleate on a substrate consisting of massive crystals of palladium metal. In contrast, the β -manganese whiskers grow at isolated sites on the bare aluminum oxide refractory. Equiaxed crystals are also present in the reaction product. The manganese silicide whiskers grow from the refractory wall and are found immersed in pools of a fused salt that flows over them as they grow. Figure 1 shows some of these whiskers after dissolution of the salt with absolute alcohol. However, it is not certain in this case whether growth takes place from the liquid or from the vapor phase (7).

The palladium whiskers occur in the form of seemingly straight rods, corkscrewlike helices, and twisted wires (see Fig. 2). Both right- and left-handed helices are observed. Frequently the pitch of the twist and the diameter of the helix or whisker change gradually along the length, and, often, abrupt changes from straight to helical form occur. The filaments growing as helices are occasionally formed from regularly connected straight segments, as illustrated by the two short helices near the center of Fig. 2. X-ray diffraction data show that the growth axis of the spiral whiskers studied is a <111> crystallographic direction. The axial direction in the straight whiskers is a high index direction varying within 12 deg around the < 211 > direction (8, 9).

The β -manganese whiskers studied



Fig. 1. Cluster of manganese silicide crystals after extraction from the remaining manganous chloride salt with ethyl alcohol. The fine isolated whisker at the lower left is 75 µ long.



Fig. 2. Portion of a cluster of palladium whiskers. Note the helices of slowly varying pitch and the abrupt transitions of pitch. Several helices are composed of connected straight segments. The length shown of the long helix is 0.8 mm.

have a <100> growth direction, and the whiskers of manganese silicide, which crystallize in a hexagonal lattice, have been observed with both <10.0>and $<00 \cdot 1>$ growth directions—that is, with growth directions either perpendicular or parallel to the basal plane.

All the palladium and manganese silicide whiskers tested could be repeatedly bent elastically to at least 2.0-percent strain, while none of the β -manganese specimens could be bent beyond about 1.5-percent elastic strain. The strains observed correspond to failure stresses of between 10^{10} and 10^{11} dy/cm² (1.5×10^{5} to 1.5×10^6 lb/in.²). Massive crystals of these materials fail at stresses smaller by several orders of magnitude.

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- A more extensive analysis of the kinetics of whisker growth by chemical reaction is being
- prepared. The assistance of R. C. Durham in the han-8. dling and mechanical testing of the whiskers, of R. D. Dragsdorf in x-ray diffraction observations, and of W. D. Forgeng in photomicrog-raphy is gratefully acknowledged. A detailed report of the dislocation configura-
- 9. tion in whiskers (including the more complex forms) is in preparation.

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