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RESEARCH LABORATORIES FOR THE ENGINEERING SCIENCES

SCHOOL OF ENGINEERING AND APPLIED SCIENCE

UNIVERSITY OF VIRGINIA

Charlottesville, Virginia 22901

A Technical Report

MICROSTRUCTURAL OBSERVATIONS ON HIGH STRENGTH POLYCRYSTALLINE IRON WHISKERS

Contract No. N00014-75-C-0691 new

Submitted to:

Office of Naval Research
Department of the Navy
800 N. Quincy Street
Arlington, VA 22217

Submitted by:

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Report No. UVA/525321/MS78/102

August 1978

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(12) 18 p.

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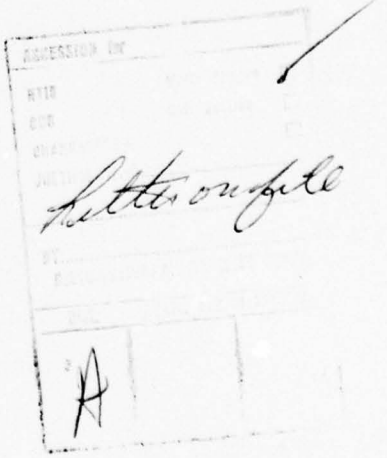
ABSTRACT

Polycrystalline iron whiskers produced by chemical vapor decomposition in the presence of a magnetic field have been investigated in regard to their microstructure and composition by transmission electron microscopy, field ion microscopy, and by X-ray and electron diffraction techniques. It was found that the whiskers consisted of a unique and complex microdispersion of iron oxides, iron carbide, and atomic carbon which bond the very small alpha-iron crystallites into a non-porous microstructure of high integrity. The mixing of strong covalent bonding with metallic bonding is proposed to explain the exceptionally high tensile strength of the whiskers.



INTRODUCTION

Lashmore et al.[1] have recently reported on the microstructure of polycrystalline iron whiskers made by the Schladitz process[2,3] where the thermal decomposition of iron pentacarbonyl in the presence of a magnetic field produces polycrystalline filaments consisting of fine grains of α-Fe 50-200 Å in diameter. In the present investigation, an attempt was made to provide additional evidence for the microstructural integrity which is thought to be responsible for the high tensile strength measured to be as high as 800 MPa. In particular, since it was recognized that overlapping diffraction contrast effects made it difficult to accurately identify individual crystalline features in the transmission electron microscope, it was decided to prepare emission end forms of the whiskers to observe the surface atomic structure by field-ion microscopy. This paper summarizes the results of a combined study employing transmission electron microscopy and field-ion microscopy. X-ray and electron diffraction data have been consulted to obtain a more definite interpretation of micrographical results.

1. Experimental

Schalidtz iron whiskers prepared as described previously[2,3] will be influenced structurally by the growth conditions which exist in the CVD reaction space. The whiskers were grown in an apparatus designed by H. Schladitz and built by his Physikalisch-Chemisches Forschungslabor, Munich, Germany. The machine was modified recently by the staff at the University of Virginia for this research by replacing the original carbonyl pump with a pressure operated carbonyl injector and by

changing the gas flow geometry in the reaction space. N_2 was used as the main carrier gas directly taken from a commercially available cylinder; the flow rate was 30 cm^3/sec. Whisker growth was initiated at 265°C and lowered within 3 minutes to 160°C which then was maintained for 6 to 30 minutes so that whiskers with different diameters could be obtained. The average magnetic field strength during these runs was 1300 Gauss.

The X-ray analysis of the whiskers was carried out with a G.E. XRD-5 diffractometer for which the whiskers were packed into a standard specimen holder; CrK_α radiation was used. Specimens for transmission electron microscopy (TEM) were obtained by placing a small bundle of whiskers between double grids. Normally, an assortment of whiskers with different diameters was present of which the majority exhibited the thickness for which the run had been designed (for example 10 μm). However, there was always some groups of whiskers of electron transparent diameter to be found which were suitable for grain size determinations and selected area diffraction. For the field-ion microscope study whiskers were separated as far as possible into single fibers approximately 0.5 cm in length, and spot welded onto 0.007 cm diameter chromel wire loops. The whiskers attached to the wire loops were then suspended in an electrolytic solution consisting of 1 part HNO_3, 1 part HCl, 4 parts H_2O, and slowly electroetched to a fine point using an a.c. voltage of 1-2 volts. The progress of the electro-polishing process was checked periodically using a metallographic microscope at 400 X.

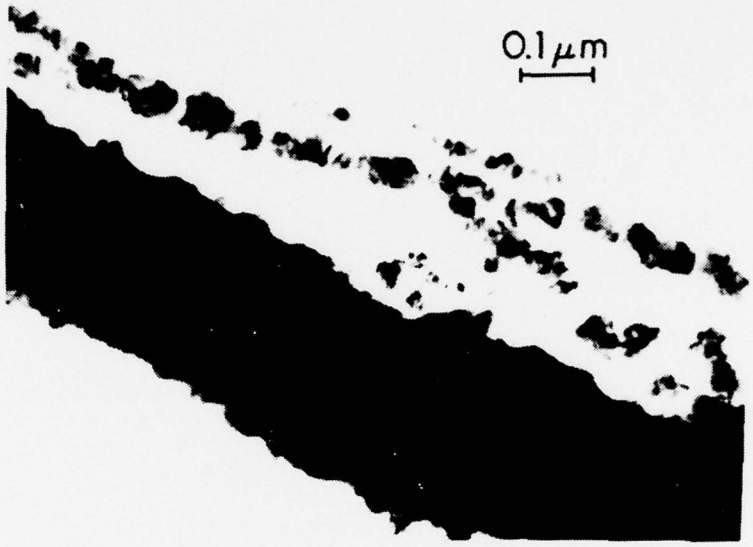
After having prepared suitable electropolished (etched) iron whisker end forms (attached to the chromel wire loops) the chromel loops were inserted into a cold-finger arrangement in a specially designed field-ion microscope and evacuated to 10^{-7} Torr. Images of the end forms were obtained using hydrogen promoted helium imaging (at a gauge pressure of roughly 10^{-4} Torr). The best images were obtained using pure neon as the imaging gas at a gauge pressure of 10^{-4} Torr. Imaging voltages were observed to range from a few Kilovolts to as high as 25 kV. A channel-plate image intensification system coupled with a Nikon-F camera, and using close-up lenses was used in recording the field-ion images of the iron whiskers. Numerous whiskers were observed prior to field-ion microscopy examination in a Hitachi Perkin-Elmer HHS-2R scanning electron microscope operated at 25 kV accelerating potential. Several whiskers were also observed prior to and following observations in the field-ion microscope in a Hitachi Perkin-Elmer H.U. 200F transmission electron microscope at an accelerating potential of 200 kV, utilizing a goniometer-tilt stage. A 500 kV RCA electron microscope and a Siemens Elmiskop IA were also used for transmission electron microscopy and selected area diffraction.

2. Results

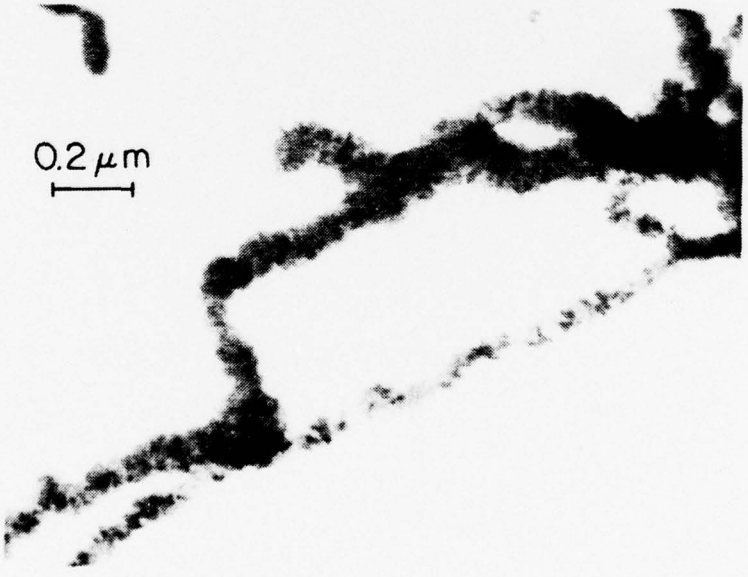
In view of the very small grain size found in polycrystalline iron whiskers, transmission electron microscopy (TEM) appears to be one of the most suitable techniques for their microstructural investigation. However, since most production runs yield whiskers thicker than 0.1 μm they are non-transparent to electrons even in high voltage electron

microscopes. This is due to multiple diffraction resulting from a number of grains which compose the whisker diameter in the direction of the incident electron beam (Fig. 1a, 1b). Thinning the whiskers for TEM by etching, electrolytical polishing or ion sputtering generally leads to uneven thicknesses which by itself poses problems in contrast interpretation. Figure 1a shows at the bottom a whisker which is nearly 0.2 μm thick. However, thinner whiskers are also visible exhibiting partial transparency and a wealth of contrast detail. The evaluation of TEM data, supported by selected area diffraction, leads to the conclusion that both, mass-thickness and diffraction contrast mechanisms, have been responsible for the details exhibited in the micrographs. The possibility of additional detail due to phase-contrast mechanisms is under study. In any event, it can be concluded without ambiguity that the individual grains are not undisturbed but contain regions below 50 Å in size which give rise to distinct contrast phenomena the details of which were not discernable by TEM.

Figure 2 illustrates the typical appearance of an iron whisker spot welded onto a chromel wire loop and electroetched to a fine-pointed emission tip. Figure 3 illustrates several typical field-ion images. These field-ion micrographs are lacking in the surface atomic symmetry and detail normally associated with field-ion images of metal and alloy emission end forms, but they are typical of the images which could be obtained using helium image-gas imaging at 78°K. The images of Figure 3 show several festures of the whisker microstructure. First, it should be apparent that there is no "long-range" crystallinity.



a) 125,000 X



b) 70,000 X

Figure 1 Transmission electron micrographs of polycrystalline iron whiskers, showing grain sizes from a few hundred angstroms to about 50 Å. Inclusions below 50 Å (not resolved) are responsible for the mottled background.

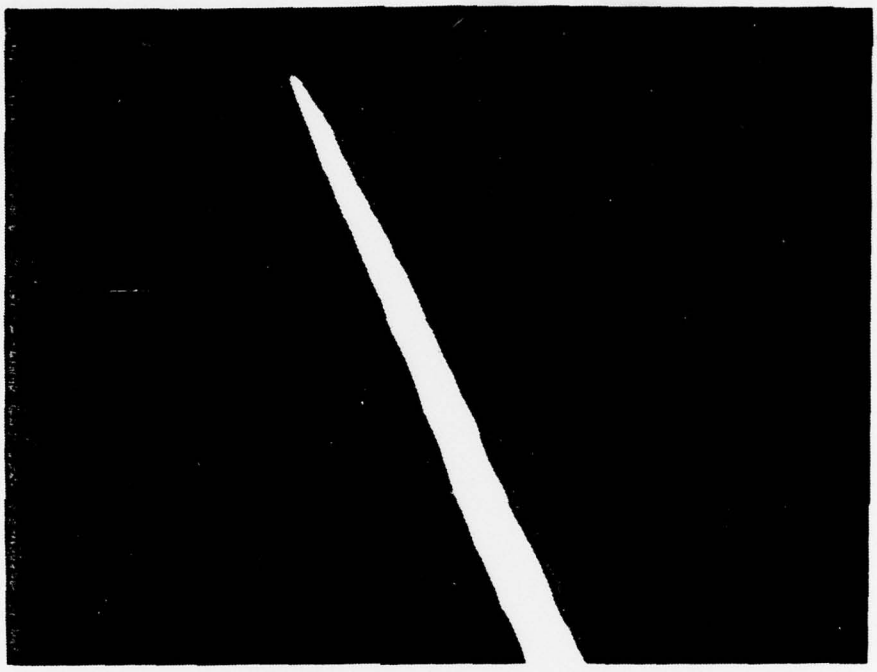
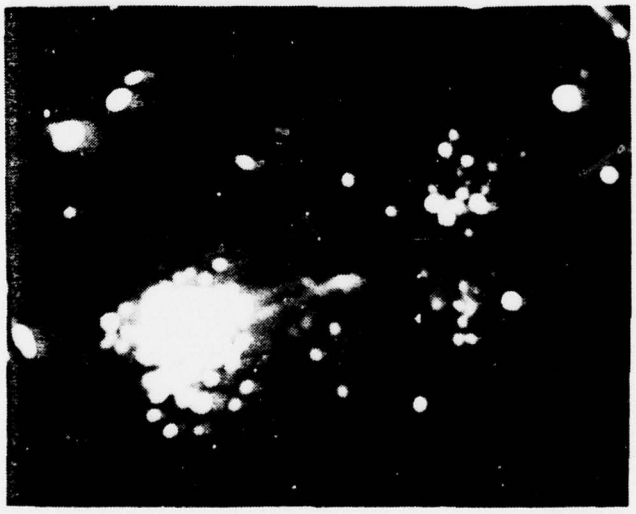


Figure 2 Scanning electron micrograph illustrating an electroetched polycrystalline iron whisker.

Figure 3(a) and (d) show that grain sizes characteristic of individual crystallites are generally about 20 Å. Furthermore, the images in Figure 3 (particularly prominent in Figure 3(a) and (b)) contain numerous, large image points which can be interpreted as being carbon or carbon-like[4], or molecular in nature, i.e. they could be carbides or oxides. Previous X-ray studies have indicated that carbon, carbides, and oxides are incorporated into the whisker microstructure[5]. Figure 3 shows that such inclusions are distributed somewhat randomly within the microstructure, and not restricted to the grain boundaries. Figure 3(c) shows the "secondary" structure of the whiskers to be concentric cylindrical shells as previously depicted by Lashmore et.al.[1]; the shells are composed of particles 1,000-2,000 Å in diameter, each particle made up of 50-100 Å iron grains. However, Figure 3(c) is from an area near the very core of the whisker and the ring displacements are only on the order of a few atomic diameter.

The fact that the field-ion images are somewhat restricted to short-range order or small crystallite areas is due in part to the fact that the minimum tip radius will be determined to a large extent by the sizes of the crystallites composing the core or center of the individual fibers. In Figure 3(c), the image is formed not only from the central core but also from a few of the concentric cylindrical shells which compose the whisker. In Figures 3(a) and (b), however, only the central core structure forms the image. This is because the electroetching produces a systematic removal of the outer surface shells as the end form or tip is created at the core. Consequently,

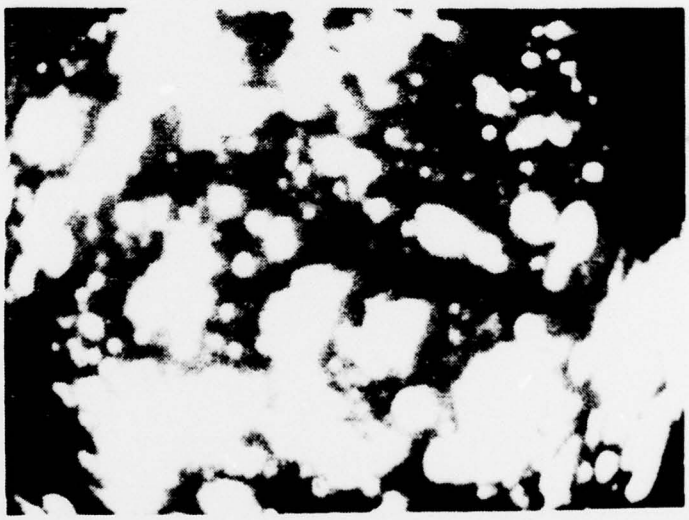
a) Edge-plane structure characteristic of small crystallite of α-Fe is shown circled. Crystal (grain) sizes measured from these observations averaged roughly 20 Å.



b) Large image spots and streaks suggestive of inclusions of carbon, carbides or oxides within the microstructure. The structure is characteristically "amorphous".

Figure 3 Field ion images (Neon images) typical of the polycrystalline iron whiskers.

c) Image showing concentric cylindrical shell structure of whiskers as described by Lashmore et al.[1] but on a much finer scale. These occur close to the core of the whisker and the rings are spaced only one or more atom diameters.



d) More developed image showing bright "islands" of α-Fe atoms in a continuous matrix of other atoms of varying identity and range of order which are, for the most part, not visible in the print shown here. The bright "islands" are not always crystalline and many areas of crystallinity are not much different from (a) insofar as size is concerned.

the images of Figures 3(a) and (b) confirm that some of the grains at the actual core are roughly 20 Å. The grain size of α-iron in the core and in the concentric cylindrical shells surrounding the core, varies between 20 Å and a few hundred angstroms depending on the local conditions governing the CVD process. Figures 3(a), (b) and (d) are illustrating this difference in the grain sizes. Figure 3(d) especially shows that the α-Fe grains are like islands dispersed in a continuum of other iron atoms and other species which may be oxides and carbides. While Figure 3(d) appears to be representative of a surface containing isolated islands of iron with voids between them, the apparent voids are not voids but simply areas of low image intensity which could not be recorded because of severe contrast restrictions. Nonetheless, with the channel plate of the field-ion microscope at nearly full intensity, the field of Figure 3(d) appeared continuous with atoms. No grain boundaries were apparent, and there was, as noted above, little crystal order in excess of regions measuring 20-50 Å in size. A great deal of the image points were unstable and images of single points (atoms or molecules) would appear, split into two arcs, grow, and disappear as if some gas or other species was being drawn from the specimen by the high field condition. Certainly the image of Figure 3(d) is not a very symmetrical array of atoms typical of an α-Fe single crystal or even polycrystalline whisker which can be obtained by field-ion microscopy, but it is representative of the structures of the whiskers examined herein. Furthermore, as can be observed from Figure 2, the whiskers prepared

for field-ion microscopy were rather symmetrically electroetched so that the end form actually imaged (Figure 3(d)) represented a region within or very near the core of the whisker.

It is known that the iron whiskers produced by the Schladitz process contain carbon and oxygen[2,5]. The carbon content has been confirmed for the whiskers made at the University of Virginia[6], which contain between 1.4 and 1.8 w/o carbon. The oxygen content has been reported by Dawihl and Eicke to be 0.8 w/o[5]. X-ray and electron diffraction patterns were obtained in order to determine in which form carbon and oxygen were present in the whiskers. Table 1 lists measurements of d-values from x-ray and electron diffraction patterns together with estimated intensities. Also, the d-values for α-Fe, α-Fe_2O_3, Fe_3O_4, Fe_3C, and graphite have been listed[7] and it is seen that a number of these substances frequently have d-spacings which coincide. X-ray and electron diffraction lines 9, 18, 20, 21 are unambiguously due to α-Fe. The presence of Fe_3O_4 and Fe_3C is clearly indicated in the x-ray diffraction data and, taking into account the overlap, the agreement with expected intensities is fair. Traces of α-Fe_2O_3 seem to be present, but graphite is not indicated.

Electron diffraction patterns hold some special interest for the analysis in that four lines are made up primarily from spots while the remaining five lines have a uniform intensity. All spotty lines are associated with α-Fe rings while lines 1, 2, 4, 15 and 17 have the d-spacings of Fe_3O_4. Although lines 4 and 15 coincide with d-spacings of Fe_3C, the intensities of the former are (100) and (85)

compared to the intensities of Fe_3C (5) and (7). Also, the strongest Fe_3C line, No. 3, is absent in the electron diffraction pattern. The spottyness of the iron rings was to be expected from the electron micrographs and agrees well with the grain size measurements of up to 300 Å. The rings ascribed to Fe_3O_4 with their homogeneous intensity, on the other hand, must be caused by crystals less than 50 Å in size.

3. Discussion and Summary

The picture which emerges for the whisker microstructure in the present investigation provides an insight which is essential for explaining the extraordinary high strength of the polycrystalline filaments. It has become clear that the microstructure is not a continuous polycrystalline regime but more appropriately characterized as a complex dispersion of fine α-Fe grains ranging in size from 20-200 Å within an assemblage of minute iron, iron oxide, iron carbide, and possibly carbon particles which in a sense bind the microstructure together. These regions, in relation to the larger crystallites of α-Fe, are approximately the same size judging from the field-ion micrographs (see Figure 3). This nearly amorphous regime is not an amorphous grain boundary region separating α-Fe, but rather a multi-phase region. Grain boundaries separating α-Fe in the sense of a well-defined interface are probably not very prominent as can be envisioned in Figure 3(d). The microstructure as emerging from this investigation also explains the lack of porosity or microporosity in the filaments.

It is the unique and complex microdispersion coupled with the very small α-Fe crystallite sizes which not only accounts for the very high strength of these whiskers but also the ability to handle them and work with them in a manner not possible with single-crystal whiskers. The incorporation of oxides, carbides, and atomic carbon into this regime might cause a mixing of strong covalent binding with the metallic binding in the iron polycrystals, and this could contribute substantially to the high strength and integrity of the total whisker microstructure.

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7. X-Ray Powder Data File, ASTM, (1960).

Table 1. X-Ray and Electron Diffraction Data from Whiskers

Ref. No.	X-ray Diffr.	d-values					Electron Diffraction
		α-Fe	Fe_3O_4	Fe_3C	α-Fe_2O_3	Graphite	
1	2	3	4	5	6	7	8
1			4.85 (40)		3.66 (25)	3.36 (100)	4.81 w cont.
2	2.98 (4)		2.97 (70)				2.99 w cont.
3	2.68 (6)				2.69 (100)		
4	2.54 (21)		2.53 (100)	2.54 (5)			2.56 m cont.
					2.51 (50)		
			2.42 (10)				
5	2.40 (12)		2.38 (65)	2.38 (65)			
6	2.28 (10)			2.26 (25)			
7	2.23 (10)			2.20 (25)			
						2.13 (10)	
8	2.10 (14)		2.09 (70)	2.10 (60)			
				2.06 (70)			
9	2.03 (st)	2.03 (100)		2.02 (60)	2.02 (30)	2.03 (50)	2.03 st spotty
				2.01 (100)			
10	1.94 (9)			1.97 (55)			
11	1.88 (12)			1.87 (30)			
12	1.86 (16)			1.85 (40)		1.80 (5)	
13	1.76 (6)			1.75 (15)			
14	1.69 (5)		1.71 (60)	1.69 (15)	1.70 (60)	1.68 (80)	
15	1.62 (7)		1.61 (85)	1.61 (7)			1.62 w cont.
16	1.59 (8)			1.58 (20)		1.54 (10)	
17	1.49 (10)		1.48 (85)		1.48 (35)		1.49 m cont.
					1.45 (35)		
18	1.43 (100)	1.43 (19)					1.43 m spotty
19	1.32 (5)		1.32 (20)				
						1.23 (30)	
20	1.17 (st)	1.17 (30)				1.16 (50)	1.16 st spotty
						1.14 (5)	
						1.12 (20)	
21	1.01 (32)	1.013 (9)					1.04 w spotty

( ) estimated intensities

w = weak int.

m = medium int.

st = strong int.

cont. = continuous rings

spotty = spots in rings

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