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EFFECTIVE SEPARATION TECHNIQUE FOR SMALL DIAMETER WHISKERS

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ABSTRACT

Alumina whiskers are highly desirable reinforcement fibers for composite materials. However, whiskers of long length and high strength must be separated from the tangled matt in which they are grown to permit fabrication of strong composites.

A successful method for separating small diameter whiskers from as-grown matt has been developed. Fifty percent by volume of the separated whiskers had an L/D ratio of 300 or greater. Room temperature tensile strengths of 540,000 N/cm^2 (783,000 psi) to 1,175,000 N/cm^2 (1,704,000 psi) were measured for separated whiskers. The separation process produced 48 percent useful whiskers.

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SUMMARY

This investigation was conducted to develop a process to separate small cross section, strong whiskers from their matt forms while retaining maximum whisker length and minimizing surface damage. Small samples of Al_2O_3 whiskers, 0.03 gms each, were separated from the as-grown matt by agitation in an aqueous solution. The separated whiskers were classified by length using a series of fine wire sieves. The weight of whiskers separated from each matt sample was determined for a range of agitation times. An average of 48 percent of the original weight of whisker matt was separated by the process developed.

The separation process effectively minimized surface damage to whiskers, as indicated by the similar tensile strength values of the as-grown and separated whiskers. The room temperature tensile strength of separated whiskers varied from 540,000 N/cm^2 (783,000 psi) to 1,175,000 N/cm^2 (1,704,000 psi) depending on the cross-sectional area of the whiskers. These tensile values were well within the scatter band for the strength of the as-grown whiskers which suggested that little or no damage occurred during separation. It was estimated that over 90 percent of all separated whiskers in this study had diameters from 0.7 to 2.0 microns. A representative length-to-diameter ratio of separated whiskers was approximately 300. Metallographic evidence suggested that defects resulting from side growths are inherent weak points in whiskers. A logical extension of these observations is that greater efforts to grow whiskers free of side growths would advance the whisker composite field.

INTRODUCTION

Fiber composite materials derive most of their strength from the reinforcing fibers. Whiskers are very small, single crystal fibers which have demonstrated mechanical properties approaching theoretical values. The potential of these fibers is reduced by the difficulty encountered in utilizing them in composites.

Whiskers are grown from a substrate and tend to form tangled clumps of fibers which are referred to as whisker matts. Individual whiskers must be removed from the matt to be used effectively. Separation is effected by introducing forces large enough to free discrete whiskers from the as-grown whisker matts. An effective whisker separation process should preserve the long length and high strength of as-grown whiskers.

Several investigators have devised methods which separate whiskers from the as-grown matt. References 1 and 2 have described the separation of metal coated whiskers by the use of air jets. This method results in a considerable amount of contact between whiskers. Numerous whisker collisions encountered in the air jet separation process would probably damage the surface of uncoated whiskers. The separation of uncoated whiskers with the use of a food blender has been reported in reference 3. Metal powder and an amount of whisker matt were agitated in a high speed food blender. This process would tend to break up whiskers into very short lengths which would be unsuitable for high temperature composite applications.

This investigation was conducted to develop a process to separate strong, small cross section whiskers from as-grown matts while retaining maximum whisker length and minimizing surface damage. Al_2O_3 whiskers were studied in this program because of their high thermodynamic stability and high temperature mechanical properties. The whiskers were classified according to whisker length. Room temperature tensile tests were performed on as-grown and separated whiskers to determine the effect of separation on whisker strength. Length-to-diameter measurements were performed on separated whiskers. Metallographic and scanning electron microscopic studies were performed on separated and unseparated whiskers to relate the as-grown defects to the effective length of separated whiskers.

MATERIALS, APPARATUS AND PROCEDURES

Materials

One hundred grams of Al_2O_3 whiskers were purchased from a commercial source for this investigation. The selection of the supplier was made after evaluating whisker matts of several commercial sources. The whisker matts were examined for whisker size and uniformity, the amount of nonwhisker debris and the relative amount of side growths observed under a high power microscope. The whiskers selected for this study appeared to have an average diameter less than two microns.

Apparatus

The apparatus used to separate the whiskers from the as-grown matt consists of a magnetic stirrer, several 250 ml Erlenmeyer flasks, and a round 2 x 3/8-inch Teflon coated spin-bar contained in the Erlenmeyer flasks.

Standard wire sieves were used to classify the separated whiskers. The screen mesh sizes of the sieves were 1000, 707, 500, 298, 177, and 125 microns.

Procedure

Separation from matt - The separation process developed in this program utilized the following procedure: Small samples, 0.03 gm each, torn from randomly selected whisker matts were separated and classified as described below. Each small sample of Al_2O_3 whiskers (0.03 gms) was placed in a 250 ml Erlenmeyer flask containing 150 ml of deionized or distilled water. A spin bar was used to stir the liquid for various times from 2 to 14 hours. The spin bar was revolved at 200 to 300 revolutions per minute. After the preselected stirring time for each sample had elapsed, the separated whiskers were classified by passing the suspension of separated whiskers through a stack of filtering sieves. The unseparated whisker clumps which collected on the 1000 micron mesh screen were weighed and then discarded. The fine debris which passed through all screens also were discarded. The separated whiskers collected on each screen were washed off each screen, filtered, and weighed. The whiskers were separated from the filter paper by burning the paper with a gas flame. The filter paper had an ash residue of less than 0.00005 gms, which was considered a negligible weight. The residue consisted of the ash and the whiskers in the form of a loosely felted matt.

Length/diameter measurements - The length-to-diameter ratio of separated whiskers was determined by the following technique. Whiskers collected on each of the screens, 707, 500, and 298 micron mesh, were mixed in a beaker of deionized water. Whiskers separated on the 177 and 125 micron screens were considered too small, from microscopic examination, and were not included in the blend. As was noted, the clumps collected on the 1000 micron screen were discarded. The whisker suspension was agitated to produce a random whisker distribution. Ten drops of the suspended whisker solution were placed in a test tube and diluted with 50 ml of deionized water. This suspended whisker solution was agitated to uniformly distribute the whiskers. One drop of this suspension was placed on a glass slide with an eye dropper. The water was evaporated from the slide leaving a random selection of separated whiskers. All of the whiskers from the drop were measured for length and cross-sectional area. Photomicrographs were taken of the cross-section of whiskers with the use of a 650X microscope and a polaroid camera attachment. All cross-sectional area measurements were determined from these photographs. The whiskers were either hexagonal or approximately square in cross section. The "effective" diameter of the square whiskers was taken as the square root of the cross-sectional area. The hexagonal whiskers were assumed to be circular and the diameter taken as the diagonal dimension. These data were used to determine the length-to-diameter of the separated whiskers.

Tensile tests - Ultimate tensile strength determinations were made of individual whiskers after separation and classification, and from selected whiskers mechanically pulled directly from the as-grown whisker matt. A commercial whisker tester, described in reference 4, was used for these tests.

Metallography - Metallographic examinations were conducted to determine the surface topography of separated whiskers. Electron photomicrographs and scanning electron photomicrographs were taken of separated whiskers showing some typical defects, such as side growths and notches produced by the removal of side growths.

RESULTS AND DISCUSSION

Separation Process

The agitation of whisker matts in aqueous suspension caused separation of the individual whiskers from the original matt form. However, portions of the whisker matt were not separated by this process. These portions were composed primarily of large whiskers or sections of whisker matt that were tightly bound together. Figure 1(a) shows a small portion of matt which was separated by six hours of stirring and figure 1(b) shows clumps of whisker matt not separated.

Weights of whiskers collected on the 1000, 298, and 125 micron screens were plotted against agitation time in figures 2, 3, and 4. The scatter in data shown in figures 2, 3, and 4 is believed to result from variations in the as-grown whisker matts. Despite the scatter, these curves show trends in the separation process.

The quantity of unseparated sections of whisker matt collected on the largest sieve, 1000 microns, was determined and plotted in figure 2. The weight of unseparated whiskers decreased steadily with increasing stirring time. The 707, 500, and 298 micron screens collected most of the useful whiskers. Figure 3 shows the results for the 298 micron screen. The trend shown here was generally similar to that obtained for the 707 and 500 micron screens. There appears to be a trend of increasing weight with increasing agitation time up to six hours. At longer agitation times, additional fracture of the longer whiskers into shorter lengths reduced the weight of whiskers collected on this screen. This decrease in whisker weight (after six hours) coincides with the increase in weight of whiskers collected on the smaller 125 micron screen, figure 4. The weight of whiskers collected on the smaller sieves increased at a fairly uniform rate for the entire range of agitiation times studied. Also, the amount of debris from the original matt, which passed through all sieves, visibly increased with agitation time.

It appears that the forces exerted on the individual whiskers, during agitation in the liquid media, were sufficient to reduce the length of the longer whiskers after a period of six hours. The increasing weight of the low L/D whiskers, shown in figure 4, could have come from two sources. They could have been present in the original matt and passed through all large screens as they were classified. Or, they may have been produced during the separation process by fracturing of some longer whiskers or by breaking off small side growths. The production of new low L/D whiskers caused the weight of short, unusable whiskers to increase steadily with separation time past six hours.

The weight of usable separated whiskers was obtained by averaging the weight of whiskers collected on screens with 707, 500, and 298 micron mesh size. An average of 48 percent of the original weight of whisker matt was collected on these screens after six to eight hours of agitation.

Length-to-Diameter Ratio

The length, cross-sectional area, diameter, L/D, and volume of each whisker measured are given in table I. Lengths ranged from 149 microns to 640 microns and cross-sectional areas from 0.29 to 4.85 square microns.

Because fiber-composite strength depends on volume percent fiber content, the determination of a representative L/D also should be made on the basis of volume, reference 5. A fiber of large L/D, for example, may be equivalent in volume to several very short L/D fibers. Plotting the data of table I, as shown in figure 5, it may be seen that 50 percent by volume of the whiskers had an L/D of about 300 or greater. This value was taken to be the representative L/D of the separated whiskers. From visual observations of many samples, it was estimated that over 90 percent of the separated whiskers had diameters between 0.7 and 2.0 microns.

Tensile Strength

The separated whiskers were tested in tension at room temperature along with the as-grown whiskers. The purpose of this test was to determine whether small diameter, uncoated whiskers could be separated and retain their inherent high strength. Figure 6 shows the tensile data of both separated and as-grown whiskers. Because they were manually plucked from the matt, most of the as-grown whiskers that were tested were rather large diameter whiskers. The separated whiskers were smaller. Both sets of data were plotted in figure 6 and a least square line drawn through each set. The strength of the as-grown whiskers varied from 134,000 N/cm^2 (194,000 psi) to 1,285,000 N/cm^2 (1,864,000 psi) while the separated whiskers varied from 540,000 N/cm^2 (783,000 psi) to 1,175,000 N/cm^2 (1,704,000 psi) depending on cross-sectional area. The strengths of the separated whiskers were within the scatter band of the as-grown whisker tensile data. These data can be used as an indication of the surface damage encountered in the separation process. Since there was little difference in strength between as-grown and separated whiskers, it is reasonable to conclude that little or no damage occurred during separation.

Metallography

Electron micrographs and scanning electron micrographs were taken to study the surface topography of individual separated whiskers. Several defects were found which resulted in a better understanding of whisker sidegrowths and the inherent notches they produce in as-grown whiskers. A notch created by separating a side growth from the main whisker body is shown in figure 7. Such notches would presumably cause stress concentrations, and would almost certainly lower the strength of the whiskers. Since the whiskers would fail preferentially at these sites, the effective length of the whisker would be the length between notches. Fortunately, most separated whiskers observed with the scanning electron microscope did not exhibit notches. The transparent nature of the side growth relative to the opaque main whisker indicates a difference in crystal orientation, figure 8. A plane of weakness would be expected at the side growth of the as-grown whisker.

The presence of side growth sites tends to limit the useful length of whiskers to that length between these sites. Since large length-to-diameter whiskers are desired, it is important to use whiskers with the lowest number of side growths possible. Research in growing whiskers with a minimum of side growths has been limited to date. Production of aluminum oxide whiskers with no side growths would constitute a distinct advance in whisker technology.

Observations on Separation Mechanism

Visual observation of the agitation of the whisker matt samples led the author to the following beliefs concerning the mechanism of whisker separation. Separation of whiskers from the matt resulted in part from shearing forces imposed by the liquid media on the matt and in part by impact of the matt with the spin bar. The round shape and relatively soft surface of the teflon coated spin bar minimized whisker damage resulting from contact with the bar. The low rotational speed of the spin bar, 200 - 300 rpm, minimized the force of impact between the whiskers and the spin bar. In addition, the deionized water acted as a cushion to reduce the force of impact between whiskers as well as between the whiskers and the spin bar.

SUMMARY OF RESULTS

The following results were obtained from an investigation to develop a process for separating small cross-section, strong whiskers from the as-grown matt while retaining maximum whisker length and minimizing surface damage:

1. A method has been developed to separate small diameter whiskers from the as-grown matt. The method consists of gently agitating the whisker matts in a solution of deionized or distilled water for times from six to eight hours. Effective separation of Al_2O_3 whiskers was attained along with the retention of high whisker strengths and relatively long whisker lengths. An average of 48 percent of the original weight of whisker matt was separated by the process developed.

2. The separation process effectively minimized surface damage to whiskers, as indicated by similar tensile strength values of as-grown and separated whiskers. The room temperature tensile strength of separated whiskers varied from 540,000 N/cm^2 (783,000 psi) to 1,175,000 N/cm^2 (1,704,000 psi) depending on the cross-sectional area of the whiskers. These tensile values were well within the scatter band for the strength of the as-grown whiskers which suggested that little or no damage occurred during separation.

3. A representative length-to-diameter ratio of separated whiskers was approximately 300. It was estimated that over 90 percent of the separated whiskers in this study had diameters between 0.7 and 2.0 microns.

4. Metallographic evidence was presented suggesting that notch defects and side growths of as-grown whiskers are inherent weak points in the whisker. Whiskers would be expected to fail at these weak points during separation or when stressed in a composite, thereby limiting the effective fiber length.

REFERENCES

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4. Cunningham, Andrew L.: Improvement of a Microtensile Machine. NASA TM X-1326, 1967.

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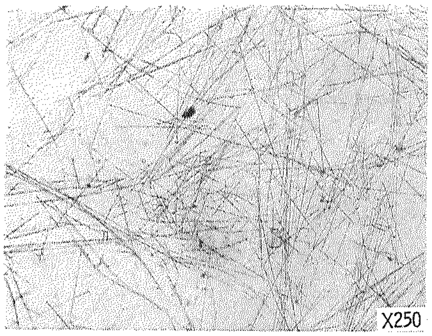
TABLE I - WHISKER PARAMETERS CALCULATED FROM LENGTH AND CROSS-SECTIONAL AREA MEASUREMENTS.

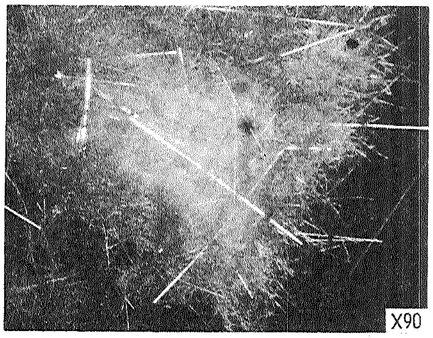
Length (microns)	Cross-Section Area (microns)^2	Diameter (microns)	L/D	Volume (microns)^3	Cummulative Volume (percent)
149	3.76	1.94	77	560	100
240	2.02	1.42	169	485	94.3
313	2.07	1.44	218	648	89.4
377	1.73	1.31	288	652	82.8
639	4.85	2.20	290	3100	76.2
356	1.48	1.21	294	527	44.2
294	0.97	0.99	297	285	38.8
640	2.70	1.64	390	1728	35.9
418	0.84	0.92	455	351	18.3
324	0.33	0.65 (Hex.)	498	107	14.7
552	0.94	0.97	569	519	13.7
451	0.48	0.78 (Hex.)	580	259	8.4
559	0.70	0.84	665	391	5.8
625	0.29	0.54	1157	181	1.8

E-6324

TYPICAL APPEARANCE OF SEPARATED AND UNSEPARATED Al_2O_3 WHISKERS



SEPARATED WHISKERS ON A GLASS SLIDE



SECTION OF WHISKER MATT NOT SEPARATED AFTER 6 HR

CS-58305

Figure 1

WHISKER WEIGHT VS AGITATION TIME

WEIGHT COLLECTED ON 1000 μM SCREEN

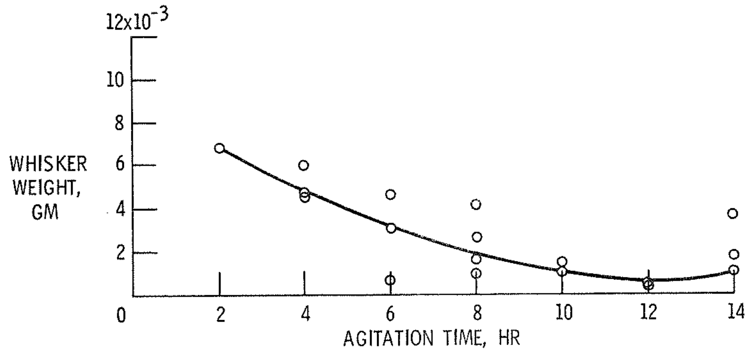


Figure 2

CS-58302

WHISKER WEIGHT VS AGITATION TIME

WEIGHT COLLECTED ON 298 μM SCREEN

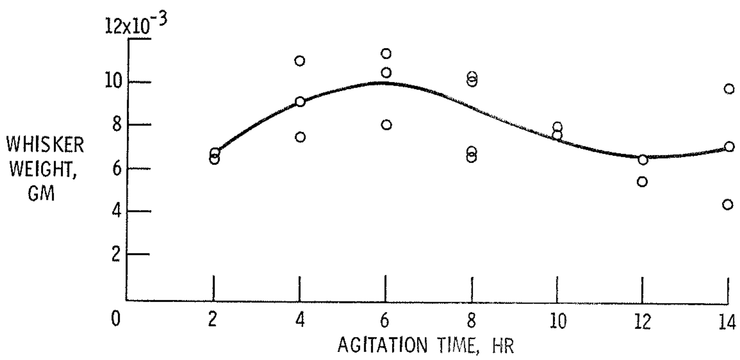


Figure 3

CS-58300

WHISKER WEIGHT VS AGITATION TIME

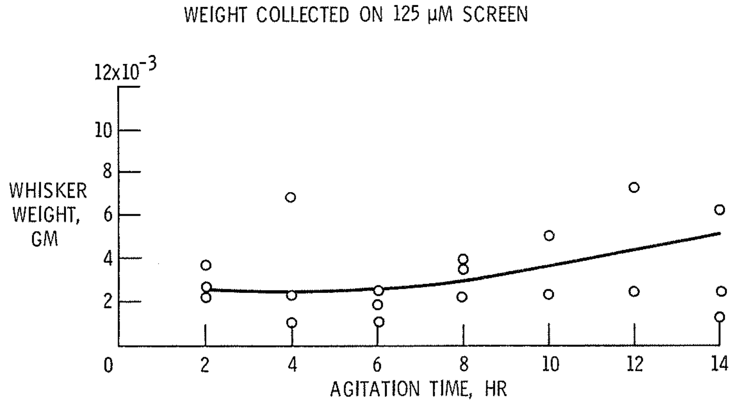


Figure 4

CUMULATIVE VOLUME PERCENT AGAINST LENGTH-TO-DIAMETER RATIO

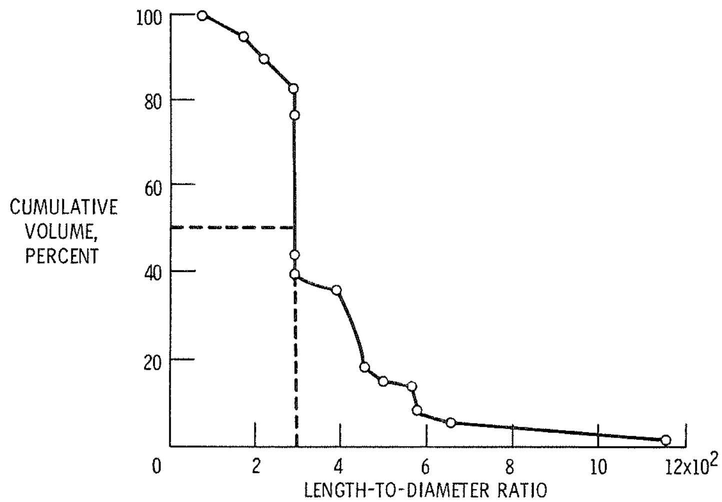


Figure 5

WHISKER TENSILE STRENGTH VS CROSS-SECTIONAL AREA

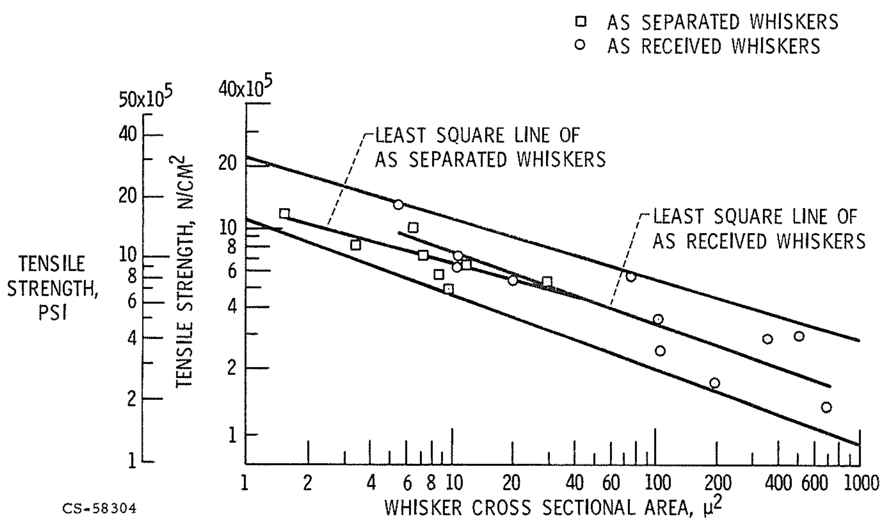


Figure 6

E-6324

SCANNING ELECTRON MICROGRAPH
OF SIDE GROWTH DEFECT ON Al_2O_3 WHISKER

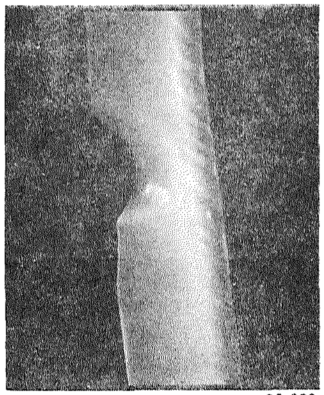


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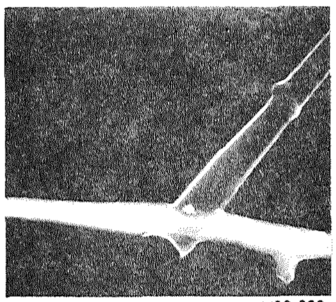
Figure 7

SCANNING ELECTRON MICROGRAPH
OF SIDE GROWTH DEFECT ON Al_2O_3 WHISKER



CS-58306 X10 000

Figure 8

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