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Preparation of thin ceramic monofilaments for TEM observation with novel embedding processes

Siwei Li [a,b], Zude Feng [a,*], Litong Zhang [b], Yanyan Wang [a], Lifu Chen [a]

[a] College of Materials, Key Laboratory of High Performance Ceramic Fibers (Xiamen University), Ministry of Education, Xiamen 361005, China
[b] National Key Laboratory of Thermostructure Composite Materials, Northwestern Polytechnical University, Xi'an 710072, China

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ABSTRACT

An applicable method to prepare transmission electron microscopy specimens from ceramic fibers for longitudinal and cross-sectional observations is investigated. The method includes novel embedding processes to fix fibers, a polishing process using a self-manufactured device to get uniformly low thickness (40 μm for L-fiber, 60 μm for C-fiber), a one-side dimpling process to grind the specimen to near electron transparency (about 5 μm in thickness for both L-fiber and C-fiber) and an efficient ion milling process using calculated parameters. These techniques are reliable to accomplish the preparation with high quality in a relatively short time. Many factors related to the preparation processes are discussed.

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1. Introduction

Specimen preparation for transmission electron microscopy (TEM) observation has long since been the limiting factor in the analysis of materials, especially for materials with special size, shape and properties [1,2]. Ceramic fibers of SiC, C, Al_2O_3, etc. have many attractive properties, i.e. high tensile strength, high hardness and good thermal stability, which make them competent for reinforcement of ceramic matrix composites (CMCs). However, diameters of these fibers are always near 10 μm; specimen preparation of the fibers for TEM observation should be technically different from that of the bulk. A reliable method to prepare thin sections of coated and uncoated ceramic fibers was reported [3], but the technique required a large amount of fibers to achieve a high fiber-to-epoxy volume ratio in a reasonably sized sample. Further, a new method was developed that worked when only a very limited amount of the fiber was available [4]. However, the improved technique required operating the embedding at least twice. After that, it also needed much time during polishing since the two glass slides used for fixing the embedded specimen needed to be ground. Besides, the polished specimen should be removed from the glass stub and remounted on a copper grid for ion milling, which was always risky since the thinned specimen (about 10 μm in thickness) was very fragile.

In this paper, practical methods with different embedding and polishing processes are investigated to prepare a TEM specimen of ceramic fiber for longitudinal observation (L-specimen) and cross-sectional observation (C-specimen). Fibers are initially fixed on a copper ring for L-specimen preparation and arranged within a copper tube for C-specimen preparation, both being embedded only once. The copper tube supports and protects the specimen throughout the whole preparation process. A simple and self-manufactured device is used to get uniform thickness instead of a tripod polisher. Calculated parameters are used in ion milling to accomplish the preparation.

2. Experimental

For preparation of the L-specimen, a Hi-Nicalon^{TM} SiC fiber (with average fiber diameter of 14 μm, Nippon Carbon, Japan) is used. Tested fibers are cut using a scalpel blade and then embedded with G1 epoxy (Gatan, USA). For preparation of C-specimen, a PAN-based carbon fiber (T300, with average fiber diameter of 7 μm, Toray, Japan) and a kind of near-stoichiometric SiC fiber (with average fiber diameter of 10 μm; not a commercial commodity; the fabrication process is reported elsewhere [5]) are selected. For embedding of the C-specimen of the near-stoichiometric SiC fiber, SiC powder is introduced as a curing additive. The volume ratios of powder–to-epoxy are selected as 0, 0.1 and 0.3. The embedded C-specimens are cut using diamond wheel saws (ISOMET^{TM}, Buehler, USA). Polishing is performed using

* Corresponding author.
E-mail address: zdfeng@xmu.edu.cn (Z. Feng).

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a self-manufactured hollow disk (the internal diameter of the disk is designed with the same size as the diameter of the glass stub used in a dimpling machine) and dimple grinding is performed by a dimpling machine (Model 656, Gatan, USA). The preparation is accomplished by ion milling using a precision ion polishing system (PIPS, Model 691, Gatan, USA). TEM observations are explored using a TECNAI F30 operated at 300 kV and JEM 2100 operated at 200 kV.

3. Specimen preparation

3.1. Embedding of L-specimen

The embedding process of L-specimen is shown in Fig. 1. A $1\times1\,cm^2$ glass slide of 1 mm thickness is cleaned in ethyl alcohol and put on a hot plate (Model 623, Gatan, USA); the upper surface of the slide is then covered by spreading thermoplastic wax as a thin layer. Subsequently, a copper ring 3 mm in external diameter is dropped flat on the melting wax before removing the glass slide from the hot plate for cooling. The wax layer coat should be as thin as possible to ensure that the copper ring is fixed but not submerged. After that, an instant adhesive is put on the two positions of the ring where the fibers will touch it. Fibers are cut to about 6 mm in length and arrayed close to each other on the adhesive. After curing (about 5 min at room temperature), the fixed fibers with the copper ring are embedded with small drops of G1 epoxy. The epoxy is then cured at 70–80 °C for 2 h. This is not a trivial matter as several factors affect the outcome of the embedding process. For example, the upper side of the copper ring should be higher than that of the wax layer. If not, the fixed fibers are embedded by the wax but not the G1 epoxy and will eventually suffer a non-protective state during heating and ion milling. Besides, the fibers must be fixed close to and parallel to each other since the overlapped fibers will be destroyed by the final grinding. The instant adhesive should not adhere to the inner ring regions of the fiber; this is to ensure a clean bond between the fiber and G1 epoxy. Finally, curing cannot be performed above 90 °C, or else the supporting wax will melt and the epoxy will attach to the glass slide; the embedded section will not be separable from the glass slide after curing.

3.2. Embedding of C-specimen

C-specimen is required in some cases for exploring the anisotropy of fibers and oriented growth of its surface coatings (or oxidized layer) induced by reaction, deposition or oxidation. The embedding process of C-specimen is shown in Fig. 2. A copper tube with an outer diameter of 3 mm is used as a holder. Two copper grids are glued on the top and bottom of the tube. Fibers are inserted into the tube within one or some of the central grids to achieve a relatively high fiber-to-epoxy volume ratio. After filling with G1 epoxy, the tube is heated at 70–80 °C for 2 h. It is worth mentioning that hardness of the epoxy is much lower than that of some of the ceramic fibers, which will induce differences in the grinding rates in different regions of the embedded specimens. Nevertheless, for the embedding of carbon fibers, G1 can be used without additives since the cured epoxy possesses a hardness similar to that of the embedded fibers, while for the SiC fiber or other ceramic fibers with much higher hardness, low-cost ceramic powders can be selected as the reinforcing agent of G1 to enhance the mean hardness of the cured epoxy and to improve the thinning quality. Different amounts of SiC powder with micron-sized particles are used in this approach using SiC as curing additives. After curing on a hot plate, the copper tube with embedded fibers is cut using a diamond wheel saw at low speeds ($\sim$100 rpm). Sections as thin as 120 μm are obtained by the proper selection of load and diamond wheel type.

3.3. Polishing

A device for obtaining uniformly low thickness is manufactured using stainless steel and used in the polishing process. As shown in Fig. 3, the hollow disk with ultra-flat surfaces has a height of 20 mm and an external diameter of 60 mm. The internal diameter of the disk is designed to be the same diameter as that of the glass stub of the dimpling machine, which ensures that the glass stub can be precisely placed into the hole and be moved in just the vertical

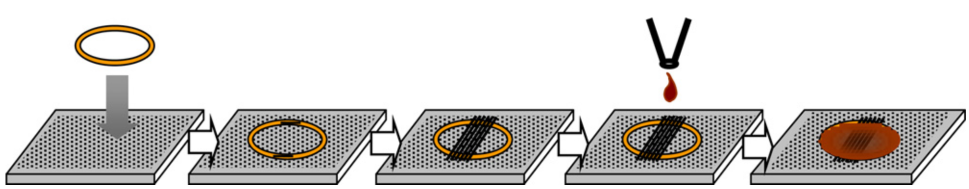

Fig. 1. Embedding process of L-specimen.

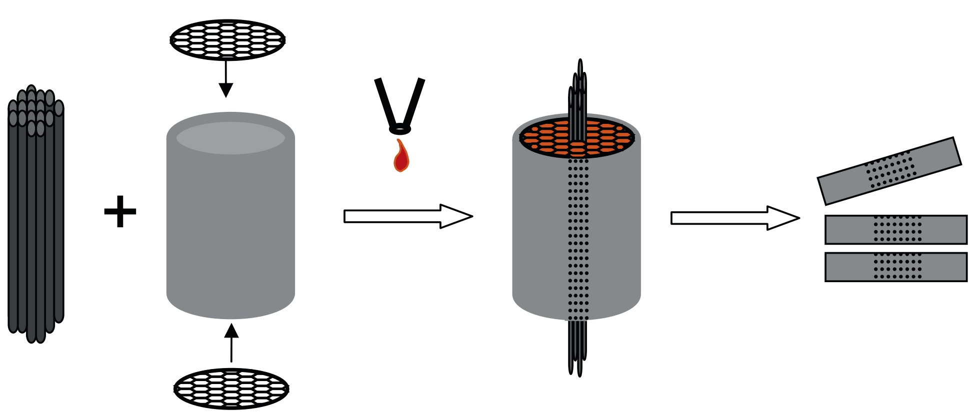

Fig. 2. Embedding process of C-specimen.

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