JOURNAL OF MATERIALS SCIENCE 21 (1986) 1809–1813

Mechanical properties and microstructure of high toughness Fe-base filaments produced by glass-coated melt spinning

TOMOKO GOTŌ, AKIHIRO YOSHINO
Department of Materials Science and Engineering, Nagoya Institute of Technology, Gokiso-cho, Showa-ku, Nagoya, Aichi, 466 Japan

High toughness $Fe_{57.5}Co_5Cr_{15}Mn_{10}Cu_2B_{10}Ti_{0.5}$ filaments having a high tensile strength of 1740 MPa and high elongation of 11.0% were produced by the method of glass-coated melt spinning. A noticeable feature of the stress–strain curves of the filament was that rapid hardening to a high stress-level of more than 1500 MPa was reached in the first few per cent of tensile elongation. The filament was 14.5 μm diameter and micropolycrystalline with a grain size of 27 nm. The crystal structure of the filament was a mixture of bcc and fcc phases and the two phases were distributed homogeneously in the filament. The high toughness of the filament related to its micropolycrystalline structure and the uniformly mixed structure of bcc and fcc phases. After heat treatment at 573 K for 600 sec, the filament developed a higher toughness with a tensile strength of 2150 MPa and an elongation of 12.0%.

1. Introduction

Glass and carbon fibres for reinforcement of composite materials are normally brittle materials with low elongation so limiting their application. High toughness metallic filaments can be employed for reinforcement of brittle matrices such as fine ceramics. We have studied the preparation of high strength and high toughness metallic filament using the method of glass-coated melt spinning [1, 2]. This method gives a filament with a cooling rate of more than $10^5\,K\,sec^{-1}$ [3] and a grain size of less than 100 nm depending on the exact alloy composition. It is possible to obtain a high toughness filament with tensile strength exceeding 2000 MPa and elongation of 10% by this method.

This paper describes the preparation of higher toughness iron-base filament and mechanical properties and microstructure of the filament obtained.

2. Experimental procedure

The melt spinning of iron-base alloys containing manganese and titanium was carried out using the same method as described earlier [1]. The parent alloys with melting points up to 1550 K were prepared by melting iron (purity 99%), boron (purity 99%) and other elements in vacuum. About 1 g alloy was placed in a Pyrex glass tube and melted by r.f. induction heating in an argon atmosphere. When the glass tube containing the molten alloy was drawn, the alloy was stretched to form a glass-coated metallic filament and was coiled on a winding drum. The glass-coating was removed in a 45% HF aqueous solution. The tensile strength of the filament produced by this method was measured with an Instron type machine at room temperature. All tests were performed with a cross-head movement of $0.0333\,mm\,sec^{-1}$ and the gauge length was 10 mm. The elongation was determined as the per cent of the displacement reached at breaking point divided by the gauge length. In these measurements, it was necessary to construct special devices because of the smallness of the filaments. The structure of the filament was examined by X-ray diffractometry.

3. Results and discussion

3.1. Preparation of high toughness iron-base filament

In a previous paper, the ductile high strength of $Fe_{67.5}Co_5Cr_{10}Ni_5Cu_2B_{10}Mo_{0.5}$ filaments with a tensile strength of 3760 MPa and an elongation of 4.8% were prepared by this method [1]. It was also found in studies of cobalt-base filament that addition of titanium and manganese elements resulted in an enhancement of toughness [2].

The melt spinning of an iron-base alloy containing titanium or manganese was therefore examined, to prepare a higher toughness filament with high elongation. The melt spinning of Fe–Co–Cr–Ni–Cu–B–Ti alloy was carried out from the molten state at a temperature of about 1600 K with winding speeds ranging from 0.95 to $7.95\,m\,sec^{-1}$. Continuous filament, which had a lustrous smooth surface and was free from pinholes, was obtained within the range of winding speeds used. The variations in average diameter and tensile strength of the filaments with winding speed were measured and the results are given in Table I. The diameter of the filament obtained decreased with increasing winding speed and ranged from 4 to 14 μm. The addition of titanium to Fe–Co–Cr–Ni–Cu–B alloy decreased the tensile strength and, to a lesser extent, elongation.

The effect of added manganese was also examined. Continuous filaments of $Fe_{64-x}Co_5Cr_{10}Ni_5Mn_5Cu_1B_{10}M_x$ (M_x = Cu, Mo, Nb, Ti and CuTi) alloys

TABLE I Spinning conditions and average tensile strengths of Fe–Co–Cr–Ni–Cu–B–Ti alloy filaments produced by melt spinning in Pyrex glass

Alloy filament	Winding speed $0.95\,m\,sec^{-1}$			Winding speed $3.97\,m\,sec^{-1}$			Winding speed $7.95\,m\,sec^{-1}$		
	Diameter (μm)	Strength (MPa)	Elongation (%)	Diameter (μm)	Strength (MPa)	Elongation (%)	Diameter (μm)	Strength (MPa)	Elongation (%)
$Fe_{69}Co_5Cr_{10}Ni_5Cu_1B_{10}$	13	1830	3.4	7.5	1180	3.6	4	1320	3.3
$Fe_{68}Co_5Cr_{10}Ni_5Cu_2B_{10}$	11	1960	3.3	4	2570	3.4	4	1990	4.1
$Fe_{73}Co_5Cr_5Ni_5Cu_2B_{9.5}Ti_{0.5}$	14	1300	2.3	5	780	2.8	4	760	2.9
$Fe_{68}Co_5Cr_{10}Ni_5Cu_2B_{9.5}Ti_{0.5}$	13	1390	3.3	7.5	1040	1.9	7	1170	1.9
$Fe_{68.5}Co_5Cr_{10}Ni_5Cu_1B_{10}Ti_{0.5}$	14	1470	3.0	–	–	–	5	1120	2.6
$Fe_{67.5}Co_5Cr_{10}Ni_5Cu_2B_7Si_3Ti_{0.5}$	12	1480	2.9	6.5	970	1.6	4.5	790	1.3

were produced by the same method. The diameter of the filament obtained decreased with increasing winding speeds and ranged from 5 to 18 μm. The average tensile strength and elongation of the filaments spun at various winding speeds were measured and are given in Fig. 1, which shows that both tensile strength and elongation increased with decreasing winding speed and that addition of copper and titanium results in an increased elongation at the lowest winding speed. For example, the $Fe_{63}Co_5Cr_{10}Ni_5Mn_5Cu_2B_{10}$ (1) and $Fe_{62.5}Co_5Cr_{10}Ni_5Mn_5Cu_2B_{10}Ti_{0.5}$ (2) filaments (1 = ● and 2 = ▲ in Fig. 1) spun at a low winding speed of $0.95\,m\,sec^{-1}$ are a ductile material having tensile strength of 1590 MPa with a high elongation of 5.5%, and a tensile strength of 1550 MPa with a high elongation of 7.3%, respectively. Although these filaments have a high elongation, their tensile strength is relatively low.

The melt spinning of $Fe_{74-x}Co_5Cr_5Mn_5Cu_1B_{10}M_x$ (M_x = Cr_5, Cr_5Mn_5 and $Cu_1Cr_5Mn_5Ti_{0.5}$) alloy was therefore carried out to prepare a filament having a high toughness. The variation with winding speed of the average tensile strength of the filament obtained is shown in Fig. 2. The tensile strength and elongation increase if extra chromium, manganese, copper and titanium are added simultaneously. A higher tensile strength of 2050 MPa with a high elongation of 7.7% is attained for the $Fe_{62.5}Co_5Cr_{10}Mn_{10}Cu_2B_{10}Ti_{0.5}$ (3) filament (3 = ● in Fig. 2) spun at the lowest winding speed of $0.95\,m\,sec^{-1}$.

Continuous filaments of $Fe_{62.5-x}Co_5Cr_{10}Mn_{10}Cu_2B_{10}Ti_{0.5}M_x$ (M_x = Si_3, Cr_5 and Mn_5) alloys were also successfully produced. The average tensile strength of the filament spun at various winding speeds is shown in Fig. 3. A higher elongation of 9.0% with high tensile strength of 1710 MPa is observed for the $Fe_{57.5}Co_5Cr_{15}Mn_{10}Cu_2B_{10}Ti_{0.5}$ (4) filament (4 = ◑ in Fig. 3) spun at the lowest winding speed of $0.95\,m\,sec^{-1}$.

3.2. Mechanical properties and microstructure of the high toughness filaments 1 to 4

The mechanical properties of the high toughness filaments 1 to 4 were measured in tension with an Instron machine. Fig. 4 shows typical examples of the apparent stress–strain curves of these filaments, which exhibited the highest strength with high elongation. A noticeable feature of the curves for filaments 3 and 4 is that rapid hardening occurs on tensile elongation and a high stress level more than 1500 MPa is reached after an elongation of a few per cent. For the filaments 1 and 2, a remarkable yield point is observed after elongation by about 1% and then the stress increases with further increase in elongation up to the breaking point. The sample of filament 4 exhibited the highest elongation of 11.1% with an apparent tensile strength of 1740 MPa. The fracture morphology of these filaments was observed by scanning electron microscopy (SEM). A representative example is shown in Fig. 5. The cup and cone type fracture is observed with minimal necking. If the filament is uniformly elongated to

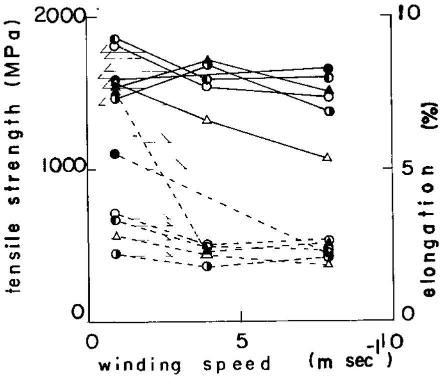

Figure 1 Average tensile strength of $Fe_{64-x}Co_5Cr_{10}Ni_5Mn_5Cu_1B_{10}M_x$ filaments spun at various winding speeds. M_x: ○ blank, ● Cu_1 (1), ◐ Mo_1, ◑ Nb_1, △ $Ti_{0.5}$, ▲ $Cu_1Ti_{0.5}$ (2).

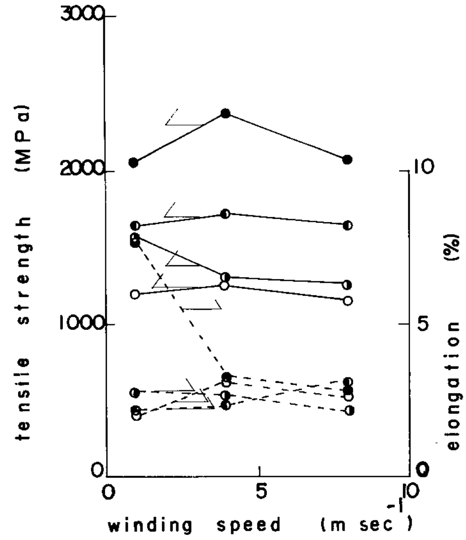

Figure 2 Average tensile strength of $Fe_{74-x}Co_5Cr_5Mn_5Cu_1B_{10}M_x$ filaments spun at various winding speeds. M_x: ○ blank, ◑ Cr_5, ● $Cu_1Ti_{0.5}Cr_5Mn_5$ (3); ◐ Cr_5Mn_5.

TABLE II Maximum toughness, crystal structure and apparent particle size of Fe–Co–Cr–Mn–Cu–B filaments 1 to 4

Alloy filament	Diameter (μm)	Toughness (MPa %)	Tensile strength (MPa)	Elongation (%)	Intensity ratio I_{fcc}/I_{bcc}	Particle size (nm)	
						bcc	fcc
(1) $Fe_{63}Co_5Cr_{10}Ni_5Mn_5Cu_2B_{10}$	15	8 500	2180	6.0	1.91	21	27
(2) $Fe_{62.5}Co_5Cr_{10}Ni_5Mn_5Cu_2B_{10}Ti_{0.5}$	18.5	9 000	1710	8.6	1.28	14	14
(3) $Fe_{62.5}Co_5Cr_{10}Mn_{10}Cu_2B_{10}Ti_{0.5}$	11	15 100	2210	8.7	0.56	31	29
(4) $Fe_{57.5}Co_5Cr_{15}Mn_{10}Cu_2B_{10}Ti_{0.5}$	14.5	16 100	1740	11.1	0.98	27	27

breaking point, the area of the sample for filament 4, shown in Fig. 4, at fracture should decrease by 11.1% compared with the initial area of the filament, and the true tensile strength of the filament is estimated to be 1930 MPa corresponding to the apparent tensile strength of 1740 MPa. The toughness of these filaments was estimated from the area of the apparent stress–strain curve shown Fig. 4. The maximum toughnesses observed are listed in Table II. The highest toughness of 16 100 MPa % was observed for filament 4.

The crystal structure of the filaments was investigated using an X-ray diffraction method. Filaments spun at the lowest winding speed of 0.95 m sec^{-1} had a mixed structure of bcc and fcc phases, whereas a single bcc phase was observed for the filaments spun at the highest winding speed of 7.95 m sec^{-1}. The proportion of the two phases was determined from the ratio of the intensity of X-ray line profile of {1 1 1} reflection of the fcc phase to that of the {1 1 0} reflection of the bcc phase and is listed in Table II. Maximum toughness and elongation were observed when this ratio was closest to unity. The proportion of fcc phase is larger for filaments 1 and 2 containing nickel than for the other filaments. Surface and cross-section of filament 4 after chemical etching in the mixture 1 g $CuSO_4$, $20 \times 10^{-6} m^3$ HCl, $20 \times 10^{-6} m^3$ CH_3CH_2OH and $20 \times 10^{-6} m^3$ H_2O were observed by SEM. The same microstructure was observed for both cross-section and surface of the filament. The surface microstructure is shown in Fig. 6. As the etching solution is characterized by tinting the austenitic phase bright [4], the fcc phase, which showed as bright spots in Fig. 6, is distributed uniformly in the filament.

Observation by transmission electron microscopy (TEM) of a filament with a cylindrical shape is very difficult because of poor transmission. In the previous paper, observation by TEM in IN 856 stainless-steel filament of 1 μm diameter was examined using a high voltage electron microscope. Only the thinnest filament of 0.25 μm prepared by electropolishing could be observed and it was found that the dislocation arrangement was so complex that individual segments of dislocation could not be observed [5]. We could not successfully thin the present filament of 14 μm diameter to 0.25 μm. Then the crystal grain size of these filaments was measured from the line profile of the X-ray diffraction pattern. The apparent particle size, ε, was determined by Scherrer's formula of $\varepsilon = \lambda/(\beta \cos \theta)$, where λ is the wavelength of the X-rays used, β is the integral line breadth, and θ is the Bragg angle. The particle sizes in the filaments were measured from the intensity profiles of the {1 1 0} reflection from the bcc phase and {1 1 1} reflection from the fcc phase and the results are shown in Table II, indicating that the filaments are micropolycrystalline with a grain size of less than 30 nm.

It is considered that the micropolycrystalline structure resulted in a decrease of dislocation pile ups at grain boundaries so dispersing the stress-concentrations associated with such pile-ups. The high toughness for the present filament is also related to the uniformly mixed structure of bcc and fcc phases, the high strength stemming mainly from the bcc phase

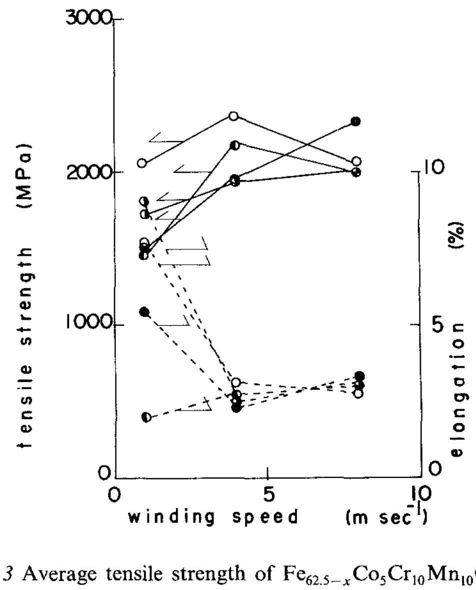

Figure 3 Average tensile strength of $Fe_{62.5-x}Co_5Cr_{10}Mn_{10}Cu_2B_{10}Ti_{0.5}M_x$ filaments spun at various winding speeds. M_x: ○ blank, ● Si_3, ◑ Cr_5 4; ◐ Mn_5.

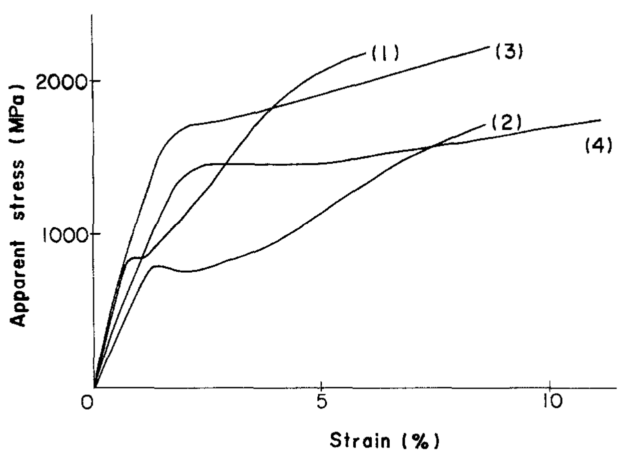

Figure 4 Apparent stress–strain curves of the high toughness filaments 1, $Fe_{63}Co_5Cr_{10}Ni_5Mn_5Cu_2B_{10}$; 2, $Fe_{62.5}Co_5Cr_{10}Ni_5Mn_5Cu_2B_{10}Ti_{0.5}$; 3, $Fe_{62.5}Co_5Cr_{10}Mn_{10}Cu_2B_{10}Ti_{0.5}$; 4, $Fe_{57.5}Co_5Cr_{15}Mn_{10}Cu_2B_{10}Ti_{0.5}$.

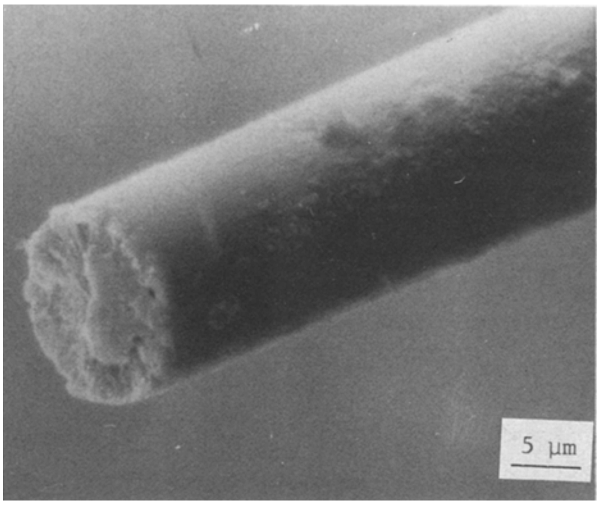

Figure 5 Fracture morphology of the $Fe_{57.5}Co_5Cr_{15}Mn_{10}Cu_2B_{10}Ti_{0.5}$ filament fractured at 1620 MPa, 9.0%.

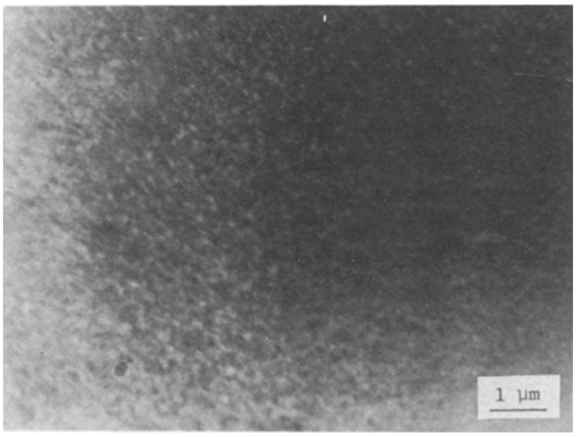

Figure 6 Scanning electron micrograph of the surface of $Fe_{57.5}Co_5$-$Cr_{15}Mn_{10}Cu_2B_{10}Ti_{0.5}$ filament after chemical etching.

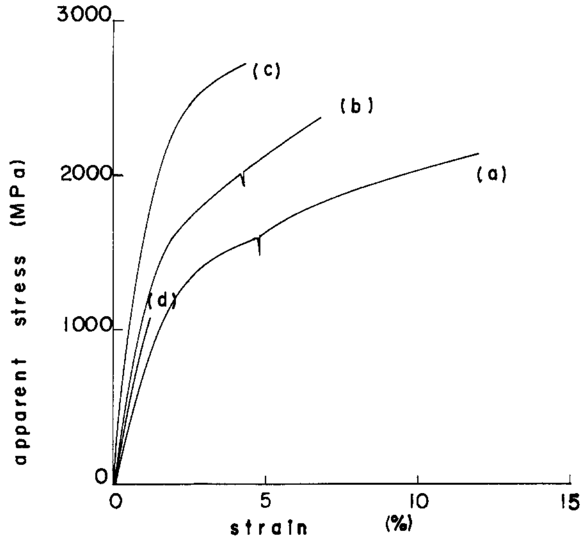

Figure 7 Apparent stress–strain curves of the $Fe_{57.5}Co_5Cr_{15}Mn_{10}$-$Cu_2B_{10}Ti_{0.5}$ filaments annealed at various temperatures for 600 sec. (a) 573 K, (b) 873 K, (c) 1073 K, (d) 1273 K.

and the ductility enhanced by the fcc phase. Thus the filaments combine high strength with high elongation.

The mechanical properties of the filament 4 annealed for 600 sec at various temperatures were also examined. The apparent stress–strain curves of the annealed filaments 4, which exhibited the highest toughness, are shown in Fig. 7. The highest elongation of 12.0% with the high tensile strength of 2150 MPa was observed for the filament annealed at 573 K for 600 sec. The elongation decreased with increasing annealing temperature. The highest strength was observed for the filament annealed at 1073 K for 600 sec. In spite of this severe heat treatment, the resulting filament was ductile with a high tensile strength of 2750 MPa and an elongation of 4.3%. The toughness of these filaments so annealed was also estimated from the area of the apparent stress–strain curves as shown in Fig. 7, and listed in Table III. The highest toughness of 19 100 MPa % was attained for the filament annealed at 573 K for 600 sec and this value is higher than that of the as-drawn filament.

The crystal structure of the filaments annealed for 600 sec at up to 973 K was a mixed structure of bcc and fcc phases, whereas the structure of the filament annealed for 600 sec at more than 1073 K was a single bcc phase. The proportion of the two phases and crystal grain size of the annealed filament were measured for the as-drawn filament and the results are also listed in Table III, showing that the crystal grain size increases with increasing annealing temperature, as would be expected.

4. Conclusion

The glass-coated melt spinning of Fe–Co–Cr–B–Mn–Ti base alloy was examined to prepare high toughness metallic filament. Continuous $Fe_{57.5}Co_5$-$Cr_{15}Mn_{10}Cu_2B_{10}Ti_{0.5}$ filament, which had a tensile strength of 1740 MPa and an elongation of 11.0% was obtained from the molten state at 1600 K for a winding speed of 0.95 m sec^{-1}. The filament was 14.5 μm diameter and was micropolycrystalline with a grain size of 27 nm. The filament had a mixed structure of bcc and fcc phases, the two phases being distributed homogeneously in the filament. The high toughness is

TABLE III Maximum toughness, crystal structure and apparent particle size of $Fe_{57.5}Co_5Cr_{15}Mn_{10}Cu_2B_{10}Ti_{0.5}$ filaments 4 annealed at various temperatures for 600 sec

Annealing temperature (K)	Toughness (MPa %)	Tensile strength (MPa)	Elongation (%)	Intensity ratio I_{fcc}/I_{bcc}	Particle size (nm)	
					bcc	fcc
As-drawn	16 100	1740	11.1	0.98	27	27
573	19 100	2150	12.0	0.55	30	27
873	12 300	2410	6.8	0.91	34	32
1073	8 600	2750	4.3	0	32	–
1273	670	1040	1.3	0	37	–

considered to arise from the microcrystalline structure and the uniformly mixed structure of bcc and fcc phases. By heat treatment for 600 sec at 573 K, the filament attained the highest toughness with a tensile strength of 2150 MPa and an elongation of 12.0%.

References

1. T. GOTŌ and T. TOYAMA, *J. Mater. Sci.* **20** (1985) 1883.
2. T. GOTŌ and A. YOSHINO, *Trans. Jpn. Inst. Metal.* **26** (1985) 557.
3. T. GOTŌ, *Seni Gakkaishi* **34** (1978) T-237.
4. Japan Institute of Metals "Metal Data Book" (Maruzen, Tokyo, 1974) p. 232.
5. T. GOTŌ, *Trans. Jpn. Inst. Metals* **19** (1978) 605.

Received 25 April
and accepted 11 July 1985