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# The preparation of ductile high strength Fe-base filaments using the methods of glass-coated melt spinning

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High toughness glass-coated metallic fibres show great promise for use in composite materials as reinforcement for brittle matrixes such as fine ceramics. This paper describes the glass-coated melt spinning of austenitic steel and Fe–B base alloys in order to prepare a ductile high strength filament. The toughness was estimated from the area of the stress–strain curves of the filament obtained. Continuous high toughness steel filament, which had a maximum toughness of 6600 MPa % with a tensile strength of 3050 MPa and an elongation of 3.1% was obtained from the molten state at 1600 K for a winding speed of 7.95 m $sec^{-1}$. The filament was $3 \times 10^{-6}$ m diameter and polycrystalline with a grain size of $1000 \times 10^{-10}$ m. The crystal structure of the filament was a single b c c phase and the phase transformed into a stable f c c structure by heat treatment at 1073 K for 600 sec. Ductile filaments of $Fe_{78-x}Co_5Ni_5Cu_2B_{10}M_x$ ($M_x$: $Cr_{5-20}$, $Cr_5Si_3$, $Cr_5Co_5$, $Cr_5Ni_5$, $Cr_{10}Mo_{0.5}$, $Cr_{10}Nb_{0.5}$) alloys were also successfully produced. The $Fe_{67.5}Co_5Cr_{10}Ni_5Cu_2B_{10}Mo_{0.5}$ filament had the highest toughness of 13 900 MPa % with tensile strength of 3760 MPa and an elongation of 4.8%. The filament had a single b c c phase.

## 1. Introduction

The demand for many kinds of reinforcements is growing steadily as a result of their increasing use in composite materials. Although the usual reinforcements such as glass fibre, carbon fibre and silicon carbide whiskers have high strengths and low densities, they are generally brittle and have a limited application.

The present authors have studied the glass-coated melt spinning of metals, whereby continuous filaments of copper, stainless-steel, superalloy of iron-base amorphous alloys finer than $10 \times 10^{-6}$ m diameter were produced directly from the melts of these metals [1–5].

High toughness glass-coated metallic fibres can be employed as a reinforcement for brittle matrix such as fine ceramics. The present method gives filament for a cooling rate of more than $10^5$ K $sec^{-1}$ and micrograins with grain size of up to $1000 \times 10^{-10}$ m depending on the exact alloy composition [1–6]. It is possible to obtain ductile high strength filaments by this method [7–9].

This paper describes the melt spinning of an iron-base alloy to prepare a ductile high strength filament, and the microstructure of the filament obtained.

## 2. Experimental procedure

The melt spinning of iron-base alloys was carried out using the same method as that for iron-base amorphous alloys [4]. The parent alloys of the iron-system with melting points of 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

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TABLE I Spinning conditions and average tensile strengths of austenitic steel filaments produced by melt spinning with Pyrex glass

| Alloy filament | Winding speed ($m\ sec^{-1}$) | Diameter ($\times 10^{-6}$ m) | Tensile strength (MPa) | Elongation (%) |
|---|---|---|---|---|
| (1) $Fe_{53}Ni_{17}Cr_{16}Cu_2B_6Si_6$ | 0.95 | 10 | 1320 | 2.6* |
| | 3.97 | 6 | 1070 | 1.9* |
| | 7.95 | 5 | 650 | 1.6 |
| (2) $Fe_{47}Ni_{17}Cr_{16}Cu_2B_6Si_2Mn_{10}$ | 0.95 | 11 | 1130 | 2.9* |
| | 3.97 | 6 | 1200 | 2.1* |
| | 7.95 | 7 | 1080 | 2.7 |
| (3) $Fe_{51}Ni_{17}Cr_{16}Cu_2B_6Si_6Mo_2$ | 0.95 | 12 | 1340 | 2.4* |
| | 3.97 | 8 | 1400 | 2.0 |
| | 7.95 | 5 | 940 | 2.2 |
| (4) $Fe_{51}Ni_{17}Cr_{16}Cu_2B_6Si_6Nb_2$ | 0.95 | 12 | 1270 | 2.2 |
| | 3.97 | 7 | 1730 | 2.3 |
| | 7.95 | 5 | 1210 | 2.4 |
| (5) $Fe_{45}Ni_{17}Cr_{16}Cu_2B_6Si_4Co_{10}$ | 0.95 | 13 | 1130 | 1.6 |
| | 3.97 | 6 | 770 | 1.4* |
| | 7.95 | 5 | 760 | 2.0* |
| (6) $Fe_{44}Ni_{17}Cr_{25}Cu_2B_6Si_6$ | 0.95 | 11 | 2260 | 3.0 |
| | 3.97 | 5 | 1970 | 2.0 |
| | 7.95 | 4 | 2310 | 3.2 |
| (7) $Fe_{39}Ni_{17}Cr_{30}Cu_2B_6Si_6$ | 0.95 | 9 | 1890 | 2.5* |
| | 3.97 | 4 | 1670 | 3.0* |
| | 7.95 | 3 | 1780 | 2.2* |

*Ductile materials.

in a 45% HF aqueous solution. The tensile strength of the filaments produced by this method was measured with an Instron type machine and the structure of the filament was examined with an X-ray diffractometer.

## 3. Results and discussion

### 3.1. Austenitic steel filament

An IN 856 filament, having a very high tensile strength of $10^4$ MPa, which increased as the diameter of the filament decreased, was produced by this method. However, the stress–strain curves of the filament exhibited linear work hardening because of the appearance of new metastable phases in the fcc austenitic phase [2, 3]. After referring to the chemical composition of IN 856 alloy of $Fe_{48.7}Ni_8Cr_{17}Cu_2Si_5B_2Mn_{17}C_{0.3}$, the parent alloy of $Fe_{53}Ni_{17}Cr_{16}Cu_2Si_6B_6$ was chosen.

The melt spinning of $Fe_{59-x-y}Ni_{17}Cr_{16}Cu_2B_6Si_yM_x$ ($Si_yM_x$: $Si_6$, $Si_2Mn_{10}$, $Si_6Mo_2$, $Si_6Nb_2$, $Si_4Co_{10}$, $Si_6Cr_9$, $Si_6Cr_{14}$) 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 filaments, which had lustrous smooth surfaces and were free from pinholes, were obtained within the range of winding speeds used. The variations in the average diameter and tensile strength of the filaments with the winding speed were measured and the results are shown in Table I.

The diameter of the filament obtained decreases with increasing winding speed and ranges from $3 \times 10^{-6}$ to $13 \times 10^{-6}$ m. The $Fe_{53}Ni_{17}Cr_{16}Cu_2Si_6B_6$ (1) filament, spun at a low winding speed of up to 3.97 m $sec^{-1}$ exhibited the range of plasticity in the stress–strain curve. The addition of manganese and chromium results in increasing tensile strength and elongation. For example, a high average tensile strength such as 1890 MPa and an elongation of 2.5% were attained for $Fe_{39}Ni_{17}Cr_{30}Cu_2B_6Si_6$ (7) filament. The effect of size on strength was not detected for the present filament, whereas the strength of IN 856 filament increased with decreasing diameter [2, 3]. The toughness of the filament with high strength was estimated from the area of the stress–strain curve. The maximum toughness is listed in Table II. A high maximum toughness of 6600 MPa% is observed for the (7) filament with tensile strength of 3050 MPa and elongation of 3.1%.

Several cross-sections of these filaments after chemical etching in the Marbel's solution were observed by scanning electron microscopy (SEM). In each case, the filaments were found to be polycrystalline. Observation by transmission electron

TABLE II Maximum toughness and crystal structures of austenitic steel filaments

| Alloy filament | Winding speed ($m\,sec^{-1}$) | Toughness (MPa %) | Tensile strength (MPa) | Elongation (%) | Crystal structure |
|---|---|---|---|---|---|
| (1) $Fe_{53}Ni_{17}Cr_{16}Cu_2B_6Si_6$ | 0.95 | 3460 | 1260 | 3.5 | f c c |
| | 7.95 | 648 | 764 | 1.6 | f c c + b c c |
| (2) $Fe_{47}Ni_{17}Cr_{16}Cu_2B_6Si_2Mn_{10}$ | 0.95 | 2370 | 1070 | 3.4 | f c c + b c c |
| | 7.95 | 1640 | 1180 | 3.1 | f c c + b c c |
| (7) $Fe_{39}Ni_{17}Cr_{30}Cu_2B_6Si_6$ | 0.95 | 3940 | 2240 | 2.9 | f c c + b c c |
| | 7.95 | 6600 | 3050 | 3.1 | b c c |

microscopy (TEM) of the filament with a cylindrical shape is very difficult because of poor electronic transmission. Moreover, a complex dislocation arrangement was observed by TEM of the IN 856 filament [2, 3] and TEM cannot be used for measurement of the crystal grain size of the present filament. Therefore, the average grain size of the filament was estimated to be $1000 \times 10^{-10}$ m by measuring the polycrystalline size on the cross-section of the filament by SEM. The grain size of the present filament is larger than that of IN 856 filament of $500 \times 10^{-10}$ m.

The crystal structure of the filament spun at various winding speeds was investigated using an X-ray diffraction method and some results are shown in Table II. The crystal structure of the (1) filament, spun at a low winding speed of 0.95 m $sec^{-1}$, is single f c c and the (7) filament spun at a high winding speed of 7.95 m $sec^{-1}$ has a single b c c phase. Thus the influence of winding speed is demonstrated for the structure of the filament. The present filament had a smooth surface and martensitic morphology was not observed [10, 11]. The dynamics of glass-coated melt spinning of copper, silver and IN 856 steel were analysed by deriving a set of simultaneous partial differential equations, which have been developed to describe the spinning of polymers [5]. It was estimated that the present method gave filaments for a cooling rate of more than $10^5$ K $sec^{-1}$: the rate was approximately proportional to the winding speed. Practically, the $Ni_{20}Fe_{60}B_{20}$ filaments of finer than $7 \times 10^{-6}$ m diameter, spun at a winding speed which was higher than 3.97 m $sec^{-1}$, had an amorphous structure. On the other hand, the $Ni_{20}Fe_{60}B_{20}$ filaments ($12 \times 10^{-6}$ m diameter), which were spun at a low winding speed of 0.95 m $sec^{-1}$, consisted of a single solution phase [6]. Then the thermal stability of the filament was examined using differential thermal analysis (DTA). A typical DTA curve of the (7) filament, spun at a high winding speed of 7.95 m $sec^{-1}$ is shown in Fig. 1. A slight exothermic peak at 560 K and a broad exothermic peak at 1020 K are observed for the as-drawn filament. These peaks were not reproduced after the first heating treatment at 1073 K.

Thermal gravity change was also measured simultaneously. No changes were observed up to 1000 K, but then the weight slightly increased and reached a 3% increase at 1073 K by the oxidation of the filament. X-ray diffraction patterns of the filament heated at 573 K showed a single b c c structure, the same as for the as-drawn filament and a single f c c structure was observed for the filament annealed at 1050 K for 600 $sec^{-1}$. These results indicate that an exothermic peak at 560 K in Fig. 1 is considered to arise from the release of energy stored during spinning and the peak at

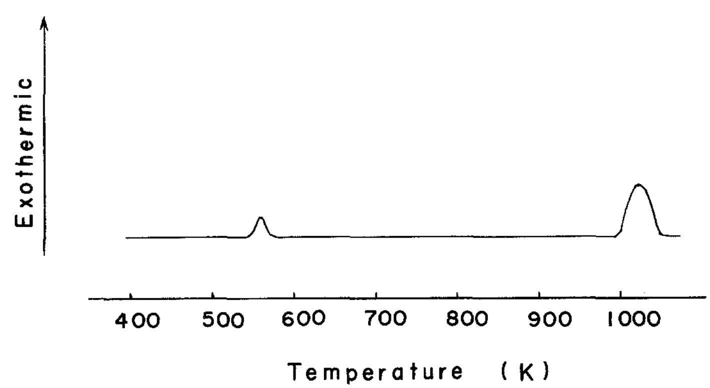


*Figure 1* DTA curve for $Fe_{39}Ni_{17}Cr_{30}Cu_2B_6Si_6$ filament spun at a winding speed of 7.95 m $sec^{-1}$ and at a heating rate of 0.334 K $sec^{-1}$.

TABLE III Spinning conditions and average tensile strengths of Fe–Co–Cr–B alloy filaments produced by melt spinning with Pyrex glass

| Alloy filament | Winding speed (m sec$^{-1}$) | Diameter ($\times 10^{-6}$ m) | Tensile strength (MPa) | Elongation (%) |
|---|---|---|---|---|
| (8) $Fe_{75}Co_5Cr_5B_{15}$ | 0.95 | 12 | 2560 | 2.5* |
| | 3.97 | 6 | 2150 | 2.8* |
| | 7.95 | 4 | 2380 | 2.8 |
| (9) $Fe_{72}Co_5Cr_5B_{15}Si_3$ | 0.95 | 9 | 2670 | 2.0* |
| | 3.97 | 5 | 2520 | 2.0 |
| | 7.95 | 4 | 2000 | 2.2 |
| (10) $Fe_{73}Co_5Cr_5B_{15}Mo_2$ | 0.95 | 12 | 2980 | 2.3 |
| | 3.97 | 5 | 3500 | 2.5 |
| | 7.95 | 4 | 3680 | 2.5 |
| (11) $Fe_{73}Co_5Cr_5B_{15}Nb_2$ | 0.95 | 16 | 1390 | 1.7* |
| | 3.97 | 6 | 1290 | 2.3* |
| | 7.95 | 3 | 1800 | 1.7* |
| (12) $Fe_{70}Co_5Cr_5B_{15}Ni_5$ | 0.95 | 13 | 2700 | 2.3* |
| | 3.97 | 8 | 1590 | 1.5* |
| | 7.95 | 3 | 2280 | 2.4* |
| (13) $Fe_{70}Co_5Cr_5B_{15}Mn_5$ | 0.95 | 12 | 2650 | 2.2 |
| | 3.97 | 7 | 1970 | 2.1 |
| | 7.95 | 4 | 1960 | 2.6 |
| (14) $Fe_{73}Co_5Cr_5B_{15}Cu_2$ | 0.95 | 11 | 1800 | 3.7* |
| | 3.97 | 7 | 1830 | 3.0* |
| | 7.95 | 6 | 1590 | 2.2* |
| (15) $Fe_{71}Co_5Cr_5B_{15}Nb_2Cu_2$ | 0.95 | 17 | 1710 | 2.1* |
| | 3.97 | 7 | 1670 | 2.5 |
| | 7.95 | 6 | 2010 | 3.0 |
| (16) $Fe_{68}Co_5Cr_5B_{15}Ni_5Cu_2$ | 0.95 | 18 | 1320 | 1.8 |
| | 3.97 | 10 | 1730 | 2.0 |
| | 7.95 | 10 | 2140 | 2.9 |

*Ductile material.

1020 K is due to the transformation of the b c c phase into the single f c c phase. The metastable phase of b c c structure formed transitionally and was transformed into the stable f c c phase by heating above 1020 K.

It is noted that a high toughness is observed for filaments consisting of a single phase.

### 3.2. Fe-base system alloy filament

It was found from studies of Fe–B base amorphous alloys that the $Fe_{76.5}Cr_{8.5}B_{15}$ and $Fe_{76.5}Co_{8.5}B_{15}$ filaments had a high tensile strength of 4000 MPa, but the stress–strain curves of these filaments did not exhibit a range of plasticity [4]. The $Fe_{75}Co_5Cr_5B_{15}$ and $Fe_{75-x}Co_5Cr_5B_{15}M_x$ ($M_x$: $Si_3$, $Mo_2$, $Nb_2$, $Ni_5$, $Mn_5$, $Cu_2$, $Cu_2Nb_2$, $Cu_2Ni_5$) filaments were produced by the same method as for the austenitic steel. The average diameter and tensile strength of the filament spun at various winding speeds are listed in Table III. The diameter of the filament obtained decreases with increasing winding speed. The stress–strain curves of the $Fe_{75}Co_5Cr_5B_{15}$ filament spun at a low winding speed of up to 3.97 m sec$^{-1}$ exhibited a range of plasticity. The tensile strength of the filament increases and plasticity decreases on addition of silicon or molybdenum. The addition of nickel, niobium and copper results in an increase in plasticity. If the two elements are added simultaneously, the range of plasticity disappears. This is considered to be caused by some metallic compounds of boron being precipitated by increasing the additional element.

Continuous filaments of $Fe_{73}Co_5Cr_5Ni_5Cu_2B_{10}$ and $Fe_{73-x}Co_5Cr_5Ni_5Cu_2B_{10}M_x$ ($M_x$: $Si_3$, $Co_5$, $Cr_5$, $Ni_5$) alloys were also successfully produced. The variation in average diameter and tensile strength of the filament with winding speed are given in Table IV. The stress–strain curve of these filaments also exhibited a range of plasticity. For example, the $Fe_{68}Co_5Cr_{10}Ni_5Cu_2B_{10}$ (20) filament is a ductile material with a high average tensile strength of 2570 MPa and a high elongation of 3.4%.

TABLE IV Spinning conditions, average tensile strengths and crystal structures of Fe–Co–Cr–Ni–Cu–B alloy filaments produced by melt spinning with Pyrex glass

| Alloy filament | Winding speed ($m\ sec^{-1}$) | Diameter ($\times 10^{-6}$ m) | Tensile strength (MPa) | Elongation* (%) | Crystal structure |
|---|---|---|---|---|---|
| (17) $Fe_{73}Co_5Cr_5Ni_5Cu_2B_{10}$ | 0.95 | 13 | 2040 | 2.1 | b c c |
| | 3.97 | 14 | 1240 | 2.1 | |
| | 7.95 | 5 | 1080 | 2.1 | b c c |
| (18) $Fe_{70}Co_5Cr_5Ni_5Cu_2B_{10}Si_3$ | 0.95 | 8 | 2550 | 2.5 | b c c + f c c |
| | 3.97 | 5 | 2740 | 2.0 | |
| | 7.95 | 4 | 2900 | 2.3 | b c c |
| (19) $Fe_{68}Co_{10}Cr_5Ni_5Cu_2B_{10}$ | 0.95 | 10 | 1730 | 2.1 | b c c |
| | 3.97 | 5 | 1370 | 2.8 | |
| | 7.95 | 3 | 1480 | 2.6 | b c c |
| (20) $Fe_{68}Co_5Cr_{10}Ni_5Cu_2B_{10}$ | 0.95 | 11 | 1960 | 3.3 | b c c |
| | 3.97 | 4 | 2570 | 3.4 | |
| | 7.95 | 4 | 1990 | 4.1 | b c c |
| (21) $Fe_{68}Co_5Cr_5Ni_{10}Cu_2B_{10}$ | 0.95 | 14 | 1550 | 3.1 | b c c |
| | 3.97 | 8 | 850 | 2.6 | |
| | 7.95 | 4 | 1070 | 3.0 | b c c |
| (22) $Fe_{63}Co_5Cr_{15}Ni_5Cu_2B_{10}$ | 0.95 | 11 | 1740 | 5.2 | b c c + f c c |
| | 3.97 | 6 | 1710 | 2.8 | |
| | 7.95 | 5 | 1790 | 2.2 | b c c |
| (23) $Fe_{58}Co_5Cr_{20}Ni_5Cu_2B_{10}$ | 0.95 | 11 | 2520 | 2.4 | b c c + f c c |
| | 3.97 | 6 | 2140 | 2.2 | |
| | 7.95 | 4 | 2400 | 2.3 | b c c |
| (24) $Fe_{65}Co_5Cr_{10}Ni_5Cu_2B_{10}Si_3$ | 0.95 | 12 | 2330 | 2.3 | b c c + f c c |
| | 3.97 | 7 | 2410 | 2.3 | |
| | 7.95 | 5 | 2150 | 2.3 | b c c |
| (25) $Fe_{67.5}Co_5Cr_{10}Ni_5Cu_2B_{10}Mo_{0.5}$ | 0.95 | 8 | 2200 | 4.0 | b c c |
| | 3.97 | 4 | 2500 | 3.5 | |
| | 7.95 | 3 | 3180 | 3.2 | b c c |
| (26) $Fe_{67.5}Co_5Cr_{10}Ni_5Cu_2B_{10}Nb_{0.5}$ | 0.95 | 12 | 1900 | 3.7 | b c c + f c c |
| | 3.97 | 6 | 3100 | 2.4 | |
| | 7.95 | 5 | 2840 | 3.2 | b c c + f c c |

*All ductile materials.

The crystal structures of the filaments spun at winding speeds of 0.95 and 7.95 $m\ sec^{-1}$, respectively, are shown in Table IV. The filaments have a single b c c structure except for the $Fe_{70}Co_5Cr_5Ni_5Cu_2B_{10}Si_3$ (18) filament spun at a low winding speed of 0.95 $m\ sec^{-1}$, which has a mixed structure of b c c and f c c phases. The DTA curve of the (18) filament spun at a winding speed of 7.95 $m\ sec^{-1}$ exhibited an irreversible exothermic peak at 963 K, whereas the peak was not detected for the (20) filament. The crystal structure of the (18) filament annealed at 1000 K for 600 sec was a mixture of f c c and b c c phases. The addition of silicon resulted in a stable structure of a mixture of b c c and f c c phases, and a single b c c phase appeared by rapid quenching.

The melt spinning of $Fe_{68-x}Co_5Cr_{10}Ni_5Cu_2B_{10}M_x$ ($M_x$: $Cr_{5,10}$, $Si_3$, $Mo_{0.5}$, $Nb_{0.5}$) was carried out to prepare a filament having a higher toughness. Variation in the average diameter, tensile strength and crystal structure of the filament with winding speed are also listed in Table IV. These filaments are ductile materials except for the filament containing silicon. The tensile strength decreased on addition of 15 at % chromium. However, a higher average elongation of 5.2% with a tensile strength of 1740 MPa is observed for the $Fe_{63}Co_5Cr_{15}Ni_5Cu_2B_{10}$ (22) filament spun at low winding speed of 0.95 $m\ sec^{-1}$. The tensile strength increases if a small amount of molybdenum or niobium is added. For example, a high average tensile strength such as 3180 MPa with an elongation of 3.2% was attained for the $Fe_{67.5}Co_5Cr_{10}Ni_5Cu_2B_{10}Mo_{0.5}$ (25) filament. The addition of silicon, niobium or chromium results in the precipitation of f c c phase in the b c c phase.

The toughness of the (20), (22) and (25) filaments with high tensile strength and high elongation

TABLE V Maximum toughness of ductile Fe–Co–Cr–Ni–Cu–B alloy filaments

| Alloy filament | Winding speed ($m\ sec^{-1}$) | Diameter ($\times 10^{-6}$ m) | Toughness (MPa %) | Tensile (MPa) | Elongation (%) |
|---|---|---|---|---|---|
| (20) $Fe_{68}Co_5Cr_{10}Ni_5Cu_2B_{10}$ | 3.97 | 3 | 9900 | 3370 | 3.5 |
| (22) $Fe_{63}Co_5Cr_{15}Ni_5Cu_2B_{10}$ | 0.95 | 12 | 9400 | 1910 | 6.8 |
| (25) $Fe_{67.5}Co_5Cr_{10}Ni_5Cu_2B_{10}Mo_{0.5}$ | 7.95 | 3 | 13 900 | 3670 | 4.8 |

was estimated from the area of the stress–strain curve. The observed maximum toughness and tensile strengths are listed in Table V. The (25) filament has the highest toughness of 13 900 MPa % and a tensile strength of 3760 MPa with an elongation of 4.8%. The filaments were also found to be polycrystalline with a grain size of $1000 \times 10^{-10}$ m.

In conclusion, continuous ductile high strength filaments of $Fe_{78-x}Co_5Ni_5Cu_2B_{10}M_x$ ($M_x$: $Cr_{5-20}$, $Cr_5Si_3$, $Cr_5Co_5$, $Cr_5Ni_5$, $Cr_{10}Mo_{0.5}$, $Cr_{10}Nb_{0.5}$) alloys could be produced from the molten state at a temperature of about 1600 K, with winding speeds ranging from 0.95 to 7.95 $m\ sec^{-1}$. The $Fe_{67.5}Co_5Cr_{10}Ni_5Cu_2B_{10}Mo_{0.5}$ filament had the highest toughness of 13 900 MPa % with a tensile strength of 3670 MPa and an elongation of 4.8%. The filaments were polycrystalline with a grain size of $1000 \times 10^{-10}$ m and had a single b c c phase.

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