Chapter 2
Fabrication of Ferromagnetic Wires

This chapter describes the fabrication methods of amorphous wires and microwires. The advantages and disadvantages of each method are examined and discussed. The existing techniques of glass removal for amorphous glass-covered wires (AGCW) are also discussed in this chapter.

2.1 Melt Spinning

Amorphous metallic alloys can be produced by a variety of rapid solidification processing techniques, including splat quenching, melt spinning, gas atomisation, and condensation from the gas phase. Among the existing techniques, the melt spinning technique has been most widely used to produce amorphous metallic alloys at cooling rates of 10^4–10^6 K/s [1]. Metallic amorphous wires are also prepared by the melt spinning method, which has been used to yield amorphous ribbons [2, 3]. The diameters of the produced wires range from 1 to 300 μm [3, 4]. The central element of this process is the pressure ejection of the melt stream through an outlet into a cooling fluid, followed by rapid solidification of this stream before it breaks into droplets. It has been highlighted that the following important conditions need to be satisfied to allow the production of metallic wires directly by the rapid solidification from the melt: (i) solidification of the metallic melt stream at high cooling rates and within the "stability" distance from the ejection point, (ii) use of a cooling fluid with low viscosity and surface tension, and (iii) stable and non-turbulent flow of the cooling liquid at high velocities. In reality, because of the difficulty of simultaneously maintaining the high supercooling capacity of the metallic melt stream without the precipitation of crystalline phases in a temperature range between the melting temperature and glass transition temperature, the melt spinning method is limited to producing metallic alloy wires with a high glass-forming ability. To overcome this problem, Ohnaka et al. [5] developed this method into the so-called in-rotating water spinning method.

H.-X. Peng et al., *Ferromagnetic Microwire Composites*,
Engineering Materials and Processes, DOI 10.1007/978-3-319-29276-2_2

2.2 In-rotating Water Spinning

This technique is modified from the melt spinning technique in that, instead of allowing the melt stream to impinge on the interior of a rotating drum, the melt stream is directly ejected into rotating water [5–7]. A cross-sectional view of an in-rotating water spinning device for producing magnetic wires is illustrated in Fig. 2.1.

It has been shown that during the in-rotating water spinning process, a jet of molten metal is ejected through a quartz nozzle of 80–200 mm diameter into a liquid cooling layer formed by a centrifugal force on the inner surface of a rotating drum of about 400–600 mm diameter. The speeds of the rotating coolant and the melt jet can be controlled by the rotation of the drum and the ejection gas pressure, respectively [6]. Depending upon the alloy being cast, it is necessary to adjust the distance between the nozzle tip and the coolant surface, the ejection angle, the depth of the coolant layer, and the coolant temperature. The in-rotating water spinning technique allows production of continuous wires of round cross section. In these wires, a dendritic structure forms along the wire direction, whereas for melt-spun ribbons, this structure tends to grow transverse to the casting direction. The cooling

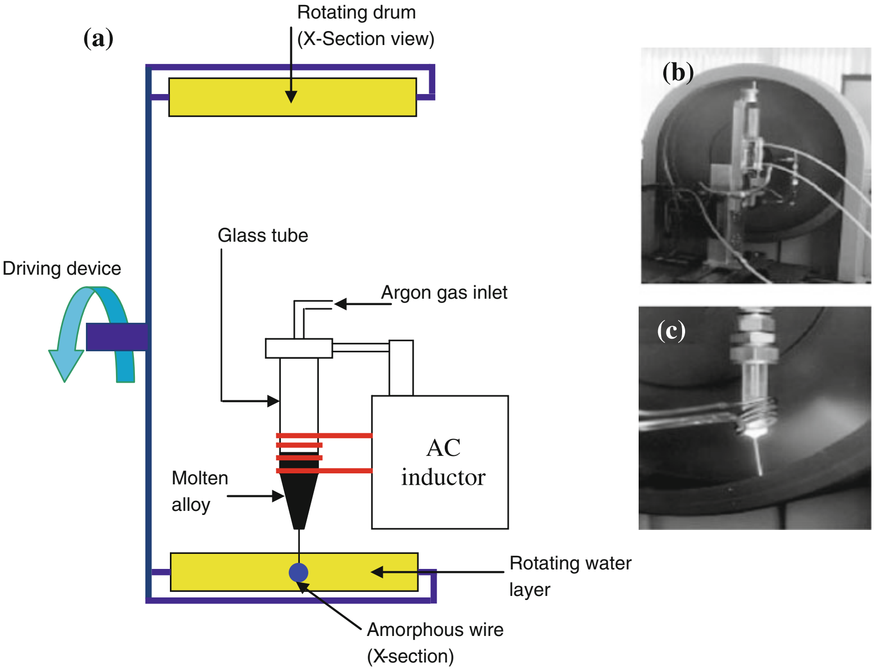

Fig. 2.1 (**a**) Cross-sectional view of the in-rotating water spinning device for producing magnetic wires with a general view (**b**) and during the melting process (**c**) (reproduced with permission from John Wiley & Sons [7])

rate is often around 10^5 K/s. A wide variety of ferrous and non-ferrous alloys have been cast into amorphous or microcrystalline wires. Amorphous metallic wires with diameters ranging from 80 to 160 μm were obtained by the in-rotating water spinning method [5]. Wires with thicker diameters of up to ∼300 μm [8], or with thinner diameters down to 30 μm [9], have also been reported. One of the main advantages of the in-rotating water spinning method is that it can be used to produce wires with alloy compositions that are difficult to obtain using the conventional methods.

2.3 Taylor-Wire Process

In 1924, Taylor [10, 11] first introduced a technique that allows the production of fine wires of uniform cross section. A schematic illustration of the Taylor-wire process can be found in [10]. In this process, a metallic charge is put in a glass tube and this material is melted by induction heating. As a result, the glass tube is softened due to its contact with the molten metal and it can then be drawn. While acting as a continuous mould crucible during solidification of the metal, it ensures a regular surface and uniform diameter of the wire. The final product consists of a metallic wire in a glass sheath and is collected on a rotating drum at speeds of around 5 m/s. The cooling rate of this process might vary in the range of 10^4–10^6 K/s for producing wires of 50 μm down to 2 μm diameter [11, 12]. A wide range of metallic wires has been produced by the Taylor method, including steels, coppers, and noble metals as well as low-melting point metals [12]. One of the main challenges of this technique is to find sheath materials that possess a sufficient chemical inertness towards the molten metal used, as well as having a softening temperature consistent with the melting temperature of the metal. However, one problem arising in this technique is the contamination of the material by the glass sheath. To avoid this problem, it is necessary to choose a glass that is compatible with the material in terms of its chemical properties, viscosity, and melting temperature. A number of recent works have evaluated the microstructure, as well as the mechanical, electrical, and magnetic properties of several microcrystalline and amorphous alloys [13].

2.4 Glass-Coated Melt Spinning

This is a modification of the Taylor-wire technique, which allows alloy systems with low wire-forming capacity to be easily produced. The ability of a metallic melt stream to break into droplets before solidification is drastically reduced by the presence of the glass covering, which prevents direct contact between the molten metal and the cooling liquid [14–19]. The glass covering ensures a smooth cylindrical shape for the melt stream. Compared with the in-rotating water spinning method, the

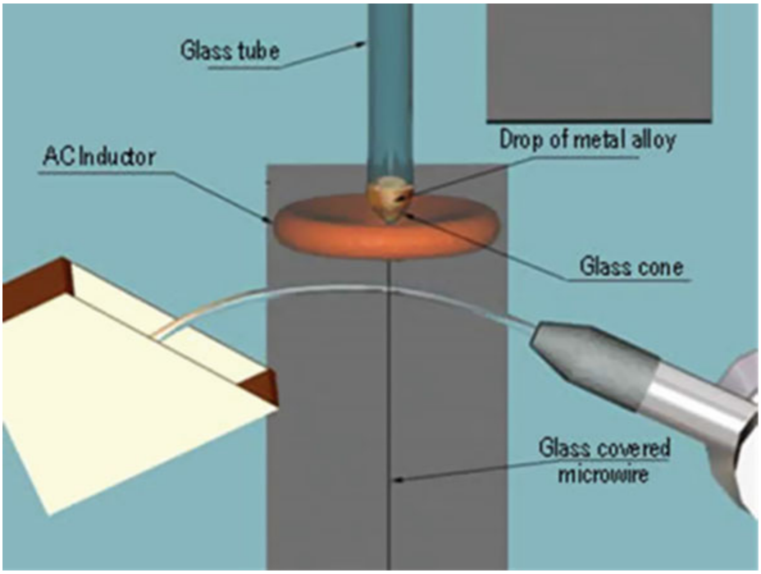

Fig. 2.2 Schematic illustration of amorphous wires/microwires fabrication process by the glass-coated melt spinning method (reproduced with permission from Elsevier [18])

glass-coated melt spinning method ensures higher cooling rates, thus producing amorphous wires more easily, even in the presence of the glass covering [18, 19].

This technique was originally proposed by Wiesner and Schneider [14] and later developed by Ulitovskiy [15, 16]. A schematic illustration of the glass-coated melt spinning method is presented in Fig. 2.2 [18]. In this process, the molten metal is captured as soon as the rapid drawing of a softened glass capillary takes place. The capillary is drawn from the end of a glass tube containing the molten metal. Alloy pieces are first put into the glass tube and then melted by a high-frequency furnace using an inductive coil. There is a softened glass covering around the molten metal drop and this allows the drawing of the capillary to take place.

To avoid any occurrence of metal oxidation, it is usual to apply a low level of vacuum of about 50–200 Pa, or an inert gas atmosphere (e.g. argon) within the glass tube. In order for the drawing process to be continuous, Chiriac et al. [17] proposed that the glass tube be displaced with a uniform speed of 0.5–7 mm/min. The as-formed wire is cooled by a water jet at approximately 1 cm under the high-frequency induction heating. Depending upon the drawing velocity, it is likely that the cooling rate varies from 10^4 to 10^7 K/s. Detailed analyses on external parameters affecting the preparation routes of wires can be found in [17–19]. Using this method, Chiriac et al. [17] produced metallic AGCW with metallic core diameters of 3–25 μm and glass thicknesses of 2–15 μm. In the case of microwires, the metallic core diameter is typically between 0.8 and 30 μm, while the thickness of the coating is in the range of 2–15 μm [18, 19]. For example, a scanning electron

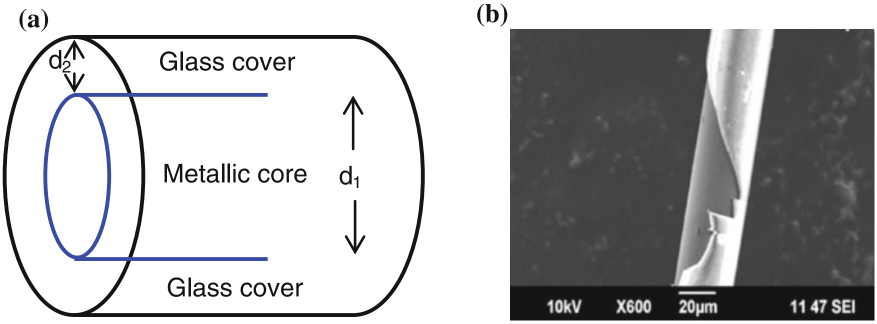

Fig. 2.3 **a** Schematic illustration of an amorphous glass-covered metallic wire. **b** Fracture surface of an amorphous $Co_{68}B_{15}Si_{10}Mn_7$ glass-covered wire

microscopy (SEM) image of a Co-rich glass-coated amorphous microwire is displayed in Fig. 2.3. Final wire dimensions such as the metallic core diameter and glass covering thickness can be accurately determined with the help of a high-resolution optical microscope engaged with a video camera and both controlled by a computer [17–19].

The glass-coated melt spinning method of wire/microwire fabrication provides several advantages over the other methods, such as (i) repeatability of wire properties at mass production and (ii) a wide range of variation in parameters (geometrical and physical), possible fabrication of continuous long wires up to 10 km and possible control and adjustment of geometrical parameters during the preparation process. Therefore, amorphous wires/microwires (Co-based and Fe-based alloys) have usually been produced by this method.

2.5 Electrodeposition

Electrodeposition is a method that has been used recently to produce uniform wires consisting of non-magnetic conducting inner core (e.g. Cu, BeCu, and W), and magnetic outer shell layers (e.g. FeNi, FeNi–Al_2O_3, and CoP) [20–36]. This method is used for coating a metallic wire with a similar or dissimilar metallic plating layer having the desired uniform thickness and a compact metallic structure by passing the wire through electrolytic baths and through surface smoothing stations. In this process, the wire is passed through an electrolytic bath to coat the wire with a plating layer and then pressed against the peripheral surfaces of rotating rollers to smooth the surface of the plating layer over the entire periphery. Finally, the wire is coated with a secondary electrolytic plating layer. The non-magnetic conducting inner cores are often wires with diameters of around 20 μm [21–24]. The magnetic layer is often thin, ranging between 2 and 7 μm. In the electrodeposition process, the thin magnetic layer is formed over the inner wire using a constant electrolytic current density. By keeping the current density constant, the layer thickness can be controlled by the deposition time. The main difficulty of this method is controlling the desired composition ratio [21–26]. This problem can be overcome by applying a longitudinal magnetic field during the deposition process, which is currently known as the magnetically controlled electroplating method [27, 28]. In addition, the application of a longitudinal magnetic field during electrodeposition results in an improvement of the uniform surface properties of the wires, which is beneficial for sensor applications [28]. In general, the electrodeposition method has the following advantages: (i) a wide range of materials (metals, alloys, and composites) that can be processed by electrodeposition is available; (ii) both continuous and batch processing are possible; (iii) materials with different grain sizes and shapes can be produced; (iv) materials with full density (i.e. negligible porosity) can be produced; and (v) the final product can be in the form of a coating or bulk material.

By combining the melt spinning and electroplating techniques, Torrejon et al. [37, 38] produced a new type of biphase magnetic microwire composed of a ferromagnetic nucleus, an intermediate glass layer, and a ferromagnetic outer shell (Fig. 2.4a). The glass-covered amorphous magnetic wires and microwires can be

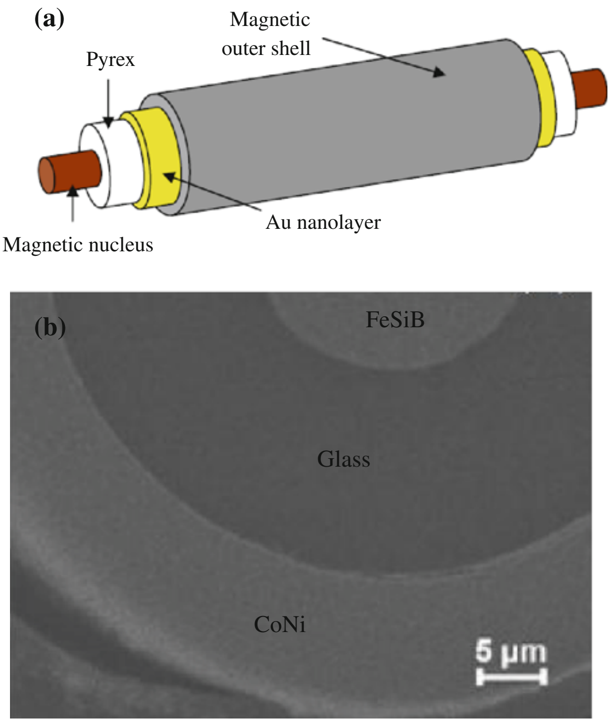

Fig. 2.4 **a** Scheme of a multilayer microwire. **b** Cross-sectional SEM image of the FeSiB/glass/Au/CoNi multilayer microwire (reproduced with permission from Elsevier [37])

obtained by the quenching and drawing method, as described above. The diameter of the nucleus and thickness of the glass covering typically range between 1 and 20 µm, respectively. Figure 2.4b displays a cross-sectional SEM image of a FeSiB/glass/Au/CoNi multilayer microwire.

A large number of different magnetic configurations such as soft/hard CoFeSiB/CoNi and FeSiB/CoNi, soft/soft CoFeSiB/FeNi, and hard/soft FePtSi/FeNi have been developed [37, 38]. It has been reported that the magnetic properties of these biphasic structures are mainly determined by the magnetic interactions between both magnetic phases, which provide the possibility of tailoring the magnetisation reversal of the soft phase through the tuning of the magnetic coupling between the two phases desirable for a number of technological applications, including multifunctional sensor devices.

2.6 Melt Extraction

Melt extraction was first applied to prepare metallic fibres as long as four decades ago [39]. It was further developed to find wide use in the fabrication of amorphous wires, ceramic fibres such as calcium aluminate [40] and high-resistivity alloy wires such as MgZnCa [41]. Recently, it has been increasingly used in the fabrication of magnetic microwires [42–46]. The basic principle of MET is to apply a high-speed wheel with a sharp edge to contact the molten alloy surface and then to rapidly extract and cool a molten layer to be wires, as schematically shown in Fig. 2.5.

There are three main advantages of this technique: (i) MET gives a higher solidification rate of 10^5–10^6 K/s than any other method, which is in favour of the form of amorphous phase. (ii) The wires produced by this method possess

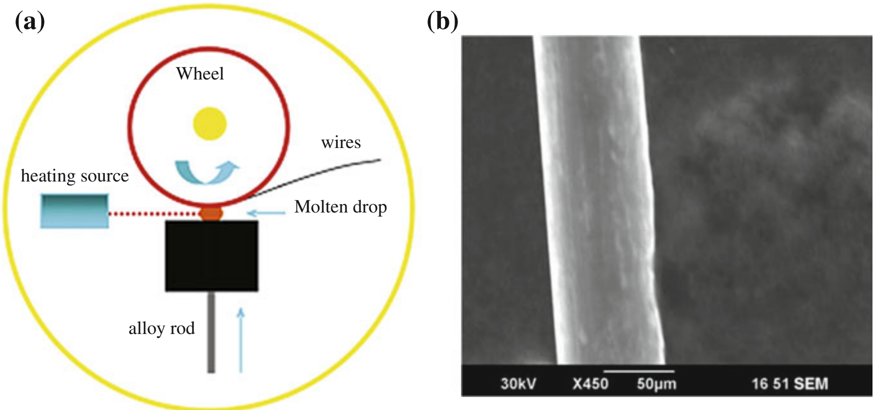

Fig. 2.5 **a** Schematic of melt extraction facility. **b** SEM image of a melt-extracted $Co_{68.2}Fe_{4.3}B_{15}Si_{12.5}$ microwire

extraordinary mechanical properties due to the high quality faultless surface and circular geometry [40, 47]. (iii) The soft magnetic properties of the materials are significantly improved by fabricating into microwires via MET; this is believed to be attributable to the considerable quenched-in stress [44]. The main drawback of this method is that it has lost control of the diameter of the produced wires. The typical diameter range for the magnetic wires is about 30–60 μm [42–44, 48] depending on the processing parameters. Apparently, it is not suitable for preparing ultrathin magnetic microwires.

2.7 Comparison of the Fabrication Technologies

In order to compare the dimensions of the wire samples produced by the above-mentioned techniques, Table 2.1 summarises the fabrication technologies and their product parameters. It is worth noting from Table 2.1 that such knowledge of the cooling rates could help us in the first instance to evaluate the relative efficiencies of the different quenching techniques. The first two methods (melt spinning and in-rotating water spinning) allow the production of metallic amorphous wires without a glass covering and are preferred for conventional amorphous wires (CAW), while the glass-coated melt spinning method (or Taylor-wire method) produces thin amorphous glass-covered wires (AGCW). From an engineering point of view, AGCW is more promising for technological applications when compared with CAW. In particular, amorphous microwires are ideal for sensing applications in microsystems [7]. The magnetic wires with glass covering are particularly useful for electrical applications in industry. Electrodeposited wires are also of interest here because of their uniform magnetic properties. However, it is quite difficult to produce such long wires using the electrodeposition technique. The synthesis of nanocrystalline wires through crystallising their amorphous precursors has been a popular method for improving the soft magnetic properties of the materials, while retaining the same dimensions of the sample. A variety of

Table 2.1 Fabrication technologies and their product parameters

Technique	Product type	Typical dimensions (μm)	Typical cooling rate (K/s)
Melt spinning	Circular section wire	1–300 diameter	10^4–10^6
In-rotating water spinning	Circular section wire	30–300 diameter	10^5–10^6
Taylor-wire process	Circular section wire	2–100 diameter	10^3–10^6
Glass-coated melt spinning	Wire/microwire with glass covering	3–50 diameter	10^4–10^7
Electrodeposition	Wire with magnetic layer	20–1000 diameter	–
Melt extraction	Amorphous wires	30–60 diameter	10^5–10^6

annealing methods (e.g. current annealing, field annealing, stress annealing, and laser annealing) have been proposed and used to produce such nanocrystalline materials. To this end, it is worth mentioning that, in the case of glass-covered wires, the removal of the glass covering may be of interest in developing specific sensing devices. The technique of glass removal is described in Sect. 2.8.

2.8 Techniques of Glass-Covering Removal

Magnetic AGCW and microwires are ideal for several applications, particularly in electrical industries, owing to the presence of the insulating glass-covered layer. In some cases, however, removal of the glass-covered layer of AGCW may be of significant interest in fundamental research as well as in practical uses, because this process can cause considerable variation in the mechanical and magnetic properties of the wire [7].

In practice, removal of the glass can be conducted either by the conventional mechanical method or by chemical etching with a hydrofluoric acid (HFA) solution. The former usually leads to a degradation of the material properties (e.g. mechanical, electrical, and magnetic) and is therefore not recommended. The latter has been shown to be a useful tool for the glass removal of AGCW, and to result in less damage. However, when the chemical etching method is used, the following features should be considered: firstly, the concentration of the HFA solution must be gradually diminished in order to avoid the etching of the metal; secondly, the last glass pieces are washed with water for the same reason. This entire process can be monitored by permanent optic control.

2.9 Concluding Remarks

The glass-coated melt spinning and single-roller melt spinning methods have been effective for preparing amorphous magnetic wires. Novel nanocrystalline wires can be obtained by annealing their corresponding amorphous alloys under optimal treatment conditions (i.e. the optimal annealing temperature and time), which have been determined based on systematic analyses by means of X-ray diffraction (XRD), differential scanning calorimetry (DSC), scanning electron microscopy (SEM), and transmission electron microscopy (TEM). The chemical method, i.e. chemical etching with a suitable HFA solution, should be employed to remove glass coating layers of magnetic wires, in order to avoid a degradation of the material properties.

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