INVESTIGATION OF THE COOLING RATE IN THE SUCTION CASTING PROCESS

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Abstract. Investigation of the cooling rate \( \varepsilon \) was carried out for suction-cast rods and tube samples of various diameters by a dendrite spacing measurements for the Fe-25 wt.% Ni alloy. Exponential change of \( \varepsilon \) with the distance \( d \) from the sample surface was observed for 2 and 4 mm dia. rods and thin walled tubes, while uniform cooling rates were measured for the 1 mm dia rods. Much larger \( \varepsilon \) values were measured for the 1 mm dia. rod and thin walled tubes.

The suction-casting technique is a method frequently used for processing bulk glassy alloys of various systems in a form of rod and tube samples [1,2]. The technique was particularly successful for producing Fe-based alloys, which require the highest cooling rates to produce amorphous samples of substantial sizes. For the Fe\(_{59}\)Co\(_{9}\)Zr\(_{10}\)Mo\(_{1}\)W\(_{2}\)B\(_{15}\) alloy [1] up to 3 mm diameter rods were possible to be cast with this technique. Furthermore, for the ferromagnetic alloys containing lower fraction of refractory elements, it was relatively easy to process 2 mm dia. amorphous rods for the soft magnetic Fe\(_{59}\)Co\(_{9}\)Zr\(_{10}\)W\(_{1}\)B\(_{15}\) alloy [2] and up to 1 mm dia rods for Fe\(_{61}\)Co\(_{13.5}\)Zr\(_{4}\)Pr\(_{4.5}\)Dy\(_{20}\) (\( x = 0, 1 \)) alloys [3]. Adjustment of an Ar pressure difference and a mass of ingot samples allowed to produce thin walled tubes of the same compositions of even larger outer diameters (o.d.) up to 4 mm (the wall thickness of \( \sim 300 \) µm). Although the glass forming ability of the alloy is of great importance, the critical cooling rate reached with the applied method is an essential factor to produce bulk glassy samples. Therefore, a goal of this work was to determine the dependence of a cooling rate \( \varepsilon \) on the diameter of the suction-cast rods and tubes. There are several methods frequently used for determining cooling rate that were systematically reviewed in [4]. The photographic technique [5] was used to determine the dependence of cooling rate on the wheel velocity and the sample section thickness for the melt-spun ribbon samples. For the piston-quenched liquid alloys the photoelectric [6] and conventional thermocouple sensors [7] were used. Further pyrometric measurements [8] allowed to find the change of cooling rate with the piston velocity. An individual character of suction-casting does not allow to apply above mentioned techniques. The major drawback of the thermocouple measurements is the time-response related to the imperfect contact with the sample. Also, this method does not allow to determine the relation between the sample section thickness and \( \varepsilon \). Pyrometric measurements are also excluded due to the error that comes out from using the arc melting of the ingot in the suction-casting technique. Also, the photographic method has to be excluded due to the radial cooling of the melt. Therefore, the only method that is suitable to determine the cooling rates for large section thicknesses seems to be a measurement of the dendrite arm spacing. It was well established that some alloy compositions can be used as a standard reference alloys to deter-
mine the cooling rates [9]. In spite of giving rather average values, this method was widely used for most of the traditional rapid solidification processes. What's more, in case of rapidly solidified rod and tube samples, this technique can allow to study the change of the cooling rate with the distance from the surface contact in the cross section of the specimens. It was shown [9] that the Fe-25Ni alloy seems to be suitable for this measurement due to a wide range of cooling rates that can be monitored (from 0.0012 to 1.7 x 10^6 K/s).

The ingot sample of the Fe – 25 wt.% Ni alloy was produced by arc-melting of the high purity elements under an Ar atmosphere. Samples were produced in a form of 1, 2, and 4 mm dia. rods and 2 and 4 mm o.d. thin walled tubes (with the wall thickness of ~300 µm for 2 mm o.d. tubes and of ~250 µm for 4 mm o.d. tubes, respectively), by the suction-casting technique. The samples were mounted into the epoxy resin and polished to observe the cross-section of the particular rods and tubes in each case. The samples were polished using classical metallographic polishing techniques to reach a smooth surface. In order to reveal dendritic microstructure on the polished surfaces, the 0.5% Nital etching was carried out for 10 s in each case. The microstructure of suction-cast rods and tubes of standard Fe-25Ni alloy was examined by metallographic microscopy. To calculate the cooling rates for particular samples, the cross-sections of the rods and tubes were photographed in high magnification. The whole area of the particular sample was divided into sections in a form of concentric rings of the constant width of 56 µm. For a particular section the cooling rate was calculated from measurements of dendrite arm spacing. The well established formula

\[ \lambda = B_6 \varepsilon^{-n}, \]  

(1)
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Fig. 2. The dependence of the cooling rates $\varepsilon$ on the distance $d$ from the die contact surface for the Fe-25Ni alloy rods of various diameters.

Fig. 3. The dependence of the cooling rates $\varepsilon$ on the distance $d$ from the die contact surface for the Fe-25Ni alloy thin walled tubes of various outer diameters.

where $n=0.32$, $B_0 = 60 \mu m/(K/s)^n$ for the Fe-25Ni alloy, $\lambda$ is dendrite arm spacing, and $\varepsilon$ – the cooling rate [9], was used for calculation of $\varepsilon$ values. The change of the cooling rate with the distance from the copper die contact surface for rods and tubes of various diameters was established for each sample.

The photographs of the representative sections variously distant from the copper die contact surface for the 4 mm dia. rod and 4 mm o.d. tube are shown in Fig. 1. It was shown that the dendrite cores lie in directions perpendicular to the rod surface in the whole area of the sample, which is the result of radial rapid solidification during suction-casting. Variation of the dendrite arm spacing was revealed on the rod cross-section. It was also shown that the dendrite arm spacing for the sections distant from the rod surface is much larger than for those
close to the surface. The calculated values of the cooling rates are of the order of $5 \times 10^3$ K/s for sections close to the rod surface and exponentially decrease to $10^2$ K/s for the areas close to the rod center (Fig. 2). The investigations were carried out up to the distance from the sample surface of $\sim 900 \mu m$, while for the central areas of the sample their different microstructure, without visible dendrites, did not allow to do the measurements. Very similar change of the cooling rates with the distance from the sample surface was observed for the 2 mm dia. rod samples also shown in Fig. 2. In this case measurements were done up to the center of the sample. Completely different results were obtained for the 1 mm dia. rods, were almost constant values of the cooling rates of $\sim 10^4$ K/s were calculated. Similar $\varepsilon$ values were obtained for the thin walled tubes, where also an exponential change of the cooling rate with the distance from the die contact surface was measured (Fig. 3).

Estimated errors were less than 10% of the $\varepsilon$ values and decreased with decrease of the cooling rates. In Figs. 2 and 3, a qualitative difference between cooling of 1 and 2 mm dia. rods and thin walled tubes was demonstrated. For tube samples the cooling rates are largest close to the surface of the samples and decrease with the distance from the surface approaching values measured for 1 mm dia rod. The most probable reason for that is a difference in the heat transfer related to unidirectional cooling in case of the tubes and radial cooling in case of rod suction-casting. Furthermore, the thickness of the samples play significant role in the cooling rates, as the rod sections near the center of the sample are not in a direct contact with the heat sink. This may impact the cooling of whole sample.

These results give a view on the possibility of processing Fe-based bulk glassy alloys. Uniform and relatively high cooling rates of processing 1 mm dia rods and thin walled tubes are the reason why it is much easier to process bulk glassy alloys in these forms.

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