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Letter to the Editor

# Microstructure and fracture surface of the two-component melt-spun amorphous/amorphous composite


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## ABSTRACT

The purpose of the research is to identify the differences in the final microstructure, fracture and short range order of the  $\text{Ni}_{55}\text{Fe}_{20}\text{Cu}_5\text{P}_{10}\text{B}_{10}$  alloy melt-spun after the ejection of the homogeneous liquid from the standard single chamber crucible and from the crucible with double chamber as the flux of two liquids i.e.: Ni–Fe–B and Ni–Cu–P. The preparation of the alloys involved remelting  $\text{Ni}_{55}\text{Fe}_{20}\text{Cu}_5\text{P}_{10}\text{B}_{10}$ ,  $\text{Ni}_{40}\text{Fe}_{40}\text{B}_{20}$  and  $\text{Ni}_{70}\text{Cu}_{10}\text{P}_{20}$  compositions in the arc furnace under titanium gettered argon atmosphere starting from pure metals 99.95 wt.% Ni, 99.95 wt.% Fe, 99.95 wt.% Cu and Ni–B, Fe–B, Ni–P, and Cu–P master alloys. The  $\text{Ni}_{55}\text{Fe}_{20}\text{Cu}_5\text{P}_{10}\text{B}_{10}$  melt-spun ribbons were obtained by means of two methods: the first one was melt-spun from a traditional single chamber crucible while the second one was produced using two component melt spinning (TCMS) performed in the protective helium atmosphere. The research was conducted by using transmission electron microscopy (TEM), scanning electron microscopy (SEM), and Mössbauer spectroscopy. The results of the microstructure examination show that the TCMS alloy is amorphous/amorphous composite and boundaries between the stripes of the differentiated chemical composition coincide with the features of the ductile fracture. The Mössbauer spectroscopy of the alloys confirms that the special feature of the TCMS alloy is that it is composed of the regions with differentiated chemical composition.

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## 1. Introduction

Metallic alloys with amorphous matrix are very promising for practical applications due to their excellent properties useful in magnetic, electrical and structural applications [1–3]. However, the use of these materials is still restricted due to their brittleness, high costs of production and limited possibility of formation amorphous/amorphous and amorphous/crystalline composites. These limitations can be removed by using a low-cost technology in the formation of amorphous/amorphous and amorphous/crystalline composites with the controllable morphology that enhances the ductility and is based on the use of low-cost precursors [4]. Amorphous/crystalline composites are obtained for systems with liquid miscibility [1,5–7]. An interesting group of metallic glasses is prepared based on liquid immiscible alloys in such systems as La–Zr–Al–Cu–Ni [8], Nd–Zr–Al–Co [9], and Ni–Nb–Y [10]. However, for these cases [8–10] the manufacturing of the two-phase metallic glasses is restricted to the alloys with a special composition. Furthermore, the microstructure of these alloys presents a large scatter of particle sizes due to liquid phase separation. The variation of the melt spinning method with the two chamber crucible (TCMS) proposed recently [11,12]

overcomes these limitations. This method enables low-cost production of an amorphous/amorphous composite with heterogeneous microstructure of the “wood-like” appearance from two glass forming melts. The purpose of this research is to show the interesting features of the  $\text{Ni}_{55}\text{Fe}_{20}\text{Cu}_5\text{P}_{10}\text{B}_{10}$  TCMS composite.

## 2. Experimental

The  $\text{Ni}_{40}\text{Fe}_{40}\text{B}_{20}$ ,  $\text{Ni}_{70}\text{Cu}_{10}\text{P}_{20}$  and  $\text{Ni}_{55}\text{Fe}_{20}\text{Cu}_5\text{P}_{10}\text{B}_{10}$  alloys were prepared starting from pure elements: 99.95 wt.% Ni, 99.95 wt.% Fe, 99.95 wt.% Cu, Ni–P, Cu–P, Ni–B, and Fe–B master alloys. The precursors were arc-melted under argon Ti gettered atmosphere. Then the alloys were melt-spun in helium atmosphere at 40 m/s and ejection pressure of 150 kPa. The crucible orifice diameter was 1.2 mm. The ribbons of the  $\text{Ni}_{55}\text{Fe}_{20}\text{Cu}_5\text{P}_{10}\text{B}_{10}$  nominal composition were obtained by ejection in two ways. The first one was obtained by two component melt-spinning (TCMS) of the  $\text{Ni}_{40}\text{Fe}_{40}\text{B}_{20}$  and  $\text{Ni}_{70}\text{Cu}_{10}\text{P}_{20}$  liquid alloys (Fig. 1). The other alloy  $\text{Ni}_{55}\text{Fe}_{20}\text{Cu}_5\text{P}_{10}\text{B}_{10}$  was melt-spun from a single-chamber. The microstructure and phase analysis of the TCMS sample was investigated using JEOL 300 kV transmission electron microscope (TEM). Cross-section of the  $\text{Ni}_{55}\text{Fe}_{20}\text{Cu}_5\text{P}_{10}\text{B}_{10}$  TCMS and fractures of the TCMS ribbon as well as  $\text{Ni}_{55}\text{Fe}_{20}\text{Cu}_5\text{P}_{10}\text{B}_{10}$  ribbon ejected from single chamber crucible after tensile test performed at strain rate 1

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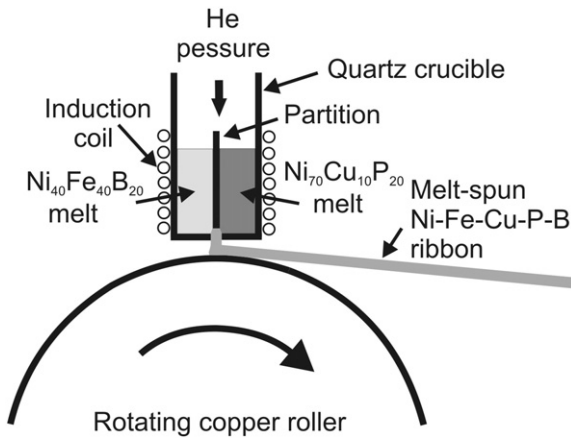


Fig. 1. The diagram showing TCMS technique [14].

mm/min were observed by means of scanning electron microscope with EDS JEOL 6610. Mössbauer transmission spectra were collected using the RENON MsAa-3 spectrometer equipped with the LND Kr-filled proportional detector. The interferometer based on He-Ne laser was used for the calibration of the velocity scale. A commercial  $^{57}\text{Co}(\text{Rh})$  source kept at room temperature was applied for 14.41-keV resonant transition in  $^{57}\text{Fe}$ . Processing of the Mössbauer data was performed using the Mosgraf-2009 software.

### 3. Results and discussion

TEM microstructure of the TCMS  $\text{Ni}_{55}\text{Fe}_{20}\text{Cu}_5\text{P}_{10}\text{B}_{10}$  alloy is presented in Fig. 2a. The microstructure of this sample forms the darker bands marked as “A” and lighter bands marked as “B” (Fig. 2a). Electron diffraction pattern presented in Fig. 2b from both areas “A” and areas “B” shows broad diffusive rings. This proves that the electron beam is scattered on the amorphous structure. These diffusive rings are located in the similar position which corresponds to the range of values between 1.9 Å and 2.3 Å. These distances are close to the values obtained for glassy Ni–P [13] and Fe–P [14], i.e.  $d = 2.02$  Å and  $d = 1.97$  Å, respectively. Differentiation of contrast between areas “A” and “B” observed in the microstructure of the TCMS alloy, may be due to the content of the species having different atomic numbers. Thus, the “A” areas are darker because they contain more Ni ( $Z = 28$ ) and Cu ( $Z = 29$ ) and “B” areas are enriched in Ni ( $Z = 28$ ) and Fe ( $Z = 26$ ).

SEM micrographs of the TCMS  $\text{Ni}_{55}\text{Fe}_{20}\text{Cu}_5\text{P}_{10}\text{B}_{10}$  alloy show the lamellar morphology that has a “wood-like” appearance consisting of the brighter and darker bands (Fig. 3). EDS line scan done across the vortex shown in Fig. 3c shows that bands have differentiated chemical composition. These areas are essentially extended along the melt spinning direction MSD. There are also frozen areas of vortices. The vortex presented in Fig. 3c is Fe-rich and its environment is enriched in Ni, Cu and P. The EDS line scan indicates that the bright areas are rich in Ni, Cu and P, and the dark areas are rich in Fe. The analysis of boron was not registered, but it is expected that the dark areas are also enriched in B. Probable explanation for the formation of a such microstructure is that the fluxes of Ni–Fe–B and Ni–Cu–P liquids were partially mixed during the passing through the hole in the crucible, however the rapid cooling during the contact of the alloy with the rotating copper roller prevented the extensive diffusion of the species and homogenization of the alloy was not possible. Comparison of these observations (Fig. 3) with the TEM results shows that these bands have glassy microstructure and the TCMS alloy is an amorphous/amorphous composite.

The fracture of the  $\text{Ni}_{55}\text{Fe}_{20}\text{Cu}_5\text{P}_{10}\text{B}_{10}$  ribbon melt-spun after ejection from the single-chamber crucible is shown in Fig. 4a, and

fracture of the TCMS  $\text{Ni}_{55}\text{Fe}_{20}\text{Cu}_5\text{P}_{10}\text{B}_{10}$  alloy with its EDS mapping is presented in Fig. 4b–f, respectively.

The fracture presented in Fig. 4a has a smooth appearance typical for a brittle glassy alloys, which is a result of intense localization of plastic flow within a single dominating shear band which is consistent with the observations of Spaepen [15]. According to these findings, the deformation of metallic glasses at low temperatures and high stresses is extremely inhomogeneous and plastic deformation is highly localized in thin shear bands. On the other hand, the fracture of the TCMS  $\text{Ni}_{55}\text{Fe}_{20}\text{Cu}_5\text{P}_{10}\text{B}_{10}$  (Fig. 4b) presents more developed surface than in the  $\text{Ni}_{55}\text{Fe}_{20}\text{Cu}_5\text{P}_{10}\text{B}_{10}$  alloy produced by using traditional single chamber crucible (Fig. 4a). The fracture surface of the TCMS  $\text{Ni}_{55}\text{Fe}_{20}\text{Cu}_5\text{P}_{10}\text{B}_{10}$  forms a ductile vein-like pattern (Fig. 4b). EDS mapping (Fig. 4c–f) reveals that there are strips of the differentiated chemical composition corresponding to the regions enriched with Ni–Fe–B and Ni–Cu–P formed during rapid freezing of the two different alloys. Furthermore, the segments of the ductile fracture coincide with the boundaries between these Ni–Fe–B and Ni–Cu–P strips (marked with dotted lines) Fig. 4c–f. The observation that the diverse composition of the Ni–Fe–B and Ni–Cu–P strips influenced fracture formation in the TCMS  $\text{Ni}_{55}\text{Fe}_{20}\text{Cu}_5\text{P}_{10}\text{B}_{10}$  alloy is in agreement with findings of Concustell et al. [16] where the Ni–Nb–Y amorphous/amorphous composite microstructure enabled the formation of multiple shear bands. The amelioration of ductility is associated with the precipitation of a second phase in the amorphous matrix. This influences the formation and paths of the shear-bands due to their disruption [16,17].

Mössbauer spectra are presented in Fig. 5, while the essential results are given in Table 1. The upper spectrum corresponds to amorphous  $\text{Ni}_{55}\text{Fe}_{20}\text{Cu}_5\text{P}_{10}\text{B}_{10}$  alloy ejected from the single chamber crucible. The

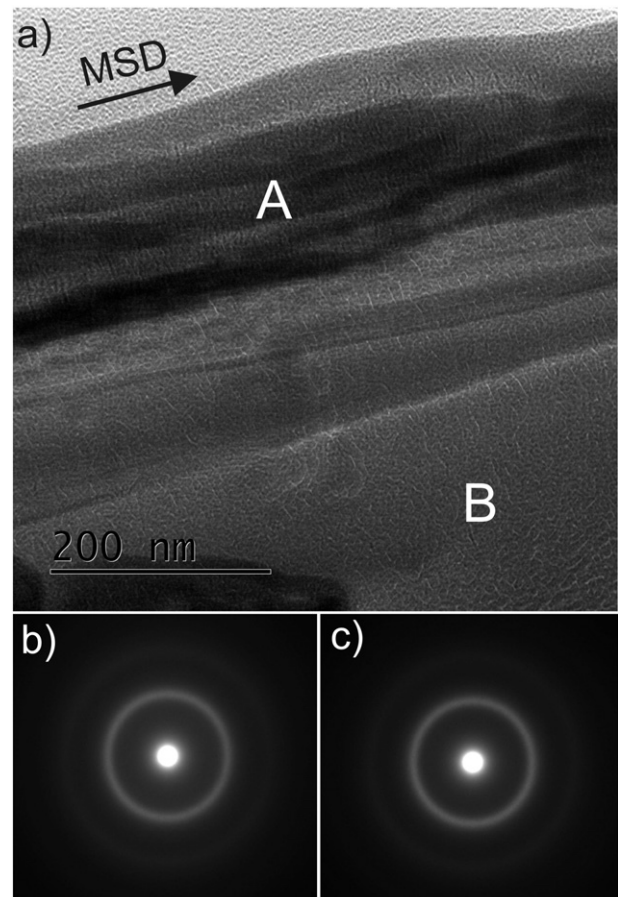


Fig. 2. TEM micrograph for the TCMS  $\text{Ni}_{55}\text{Fe}_{20}\text{Cu}_5\text{P}_{10}\text{B}_{10}$  ribbon; a) TEM image with the darker areas marked as “A” and the brighter areas marked as “B”; b) electron diffraction pattern from area “A”; c) electron diffraction pattern from area “B”.

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