

# A study of glass-formation, formation of the supercooled liquid and devitrification behavior of Ni-based bulk glass-forming alloys

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## Abstract

Glass-formation and devitrification of the glassy phase in the Ni–Nb–Zr–Ti and Ni–Nb–Zr–Ti–NM (NM, noble metals) system alloys exhibiting the supercooled liquid on heating were investigated. The crystallized structure and kinetics of devitrification of Ni–Nb–Zr–Ti–Pd glassy alloy are studied in detail. The formation of the glassy single phase has been proven using high-resolution transmission electron microscopy (TEM). The addition of Zr and Pd increases the supercooled liquid region of the Ni<sub>60</sub>Nb<sub>25</sub>Ti<sub>15</sub> (numbers indicate at.%) alloy from 44 K to about 50 K. The cP2 TiNi phase was found to form during the first exothermic phase transformation in all studied alloys. The addition of Zr to Ni–Nb–Ti alloy prevents formation of oP8 Ni<sub>3</sub>Nb phase observed in the Ni<sub>60</sub>Nb<sub>25</sub>Ti<sub>15</sub> alloy and initiates formation of the primary Ni<sub>10</sub>Zr<sub>7</sub> phase while the additions of both NM and Zr effectively refine cP2 TiNi particles size. Definite fractions of rhombohedral R and hR14 Ni<sub>4</sub>Ti<sub>3</sub> phases were also observed in NM-bearing alloys. Significantly lower growth rates of the devitrification products in the NM-bearing alloys compared to the Ni–Nb–Ti one may be the reason for the improvement of glass-forming ability (GFA).

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## 1. Introduction

Bulk glassy alloys with a size exceeding 1 mm (maximum 100 mm) in three dimensional space were obtained by stabilization of the supercooled liquid against crystallization in the various, mostly multi-component, metallic alloys [1–3]. These alloys are promising materials for structural applications as they exhibit high mechanical strength, high hardness, good fracture toughness, good corrosion resistance and so on [2]. In some alloys partial devitrification of a glassy phase forming nanocrystalline or nanoquasicrystalline precipitates is known to cause its ductilization at definite volume fraction of the precipitates [2].

Recently, bulk glass-formation was achieved in various Ni-base alloy systems: Ni–Zr–Ti–(Si, Sn) [4,5], Ni–Nb–Ti–Zr [6], Ni–Nb–Ti [7], Ni–Nb–Ti–Hf [8,9], Ni–Nb–Sn [10] and so on. They belong to an LTM–ETM or LTM–ETM–M group of alloys (where LTM is(are) late transition metal(s), ETM is(are) early transition metal(s) and M metalloids or Sn). Ni<sub>60</sub>Nb<sub>25</sub>Ti<sub>15</sub>

(numbers indicate at.%) bulk glassy alloy showed high ultimate compressive strength of 3085 MPa and plastic deformation of about 2% [7]. Multicomponent Ni–Nb–Ti–Zr–Co–Cu bulk glassy alloy with a critical diameter of 3 mm exhibited high tensile fracture strength of 2700 MPa [11]. Surprisingly Ni<sub>59</sub>Zr<sub>16</sub>Ti<sub>13</sub>Si<sub>3</sub>Sn<sub>2</sub>Nb<sub>7</sub> bulk glassy alloy, produced recently, showed a significant plastic deformation of 6.5% to failure [12]. It allows to suggest that this alloy may contain nanoparticles which are not detectable by an X-ray diffraction (XRD) technique.

It has been also shown that an addition of Ni to Cu-based LTM–ETM glass-forming alloys significantly changes their devitrification behavior [13]. Also contrary to Cu<sub>55</sub>Zr<sub>30</sub>Ti<sub>10</sub>Pd<sub>5</sub> alloy [14,15] no icosahedral phase has been observed in Ni<sub>55</sub>Zr<sub>30</sub>Ti<sub>10</sub>Pd<sub>5</sub> one [16].

Devitrification (crystallization) behavior of Ni<sub>60</sub>Nb<sub>25</sub>Ti<sub>15</sub> glassy alloy has been studied recently [17]. It has been also found that the addition of noble metals (NM) improves the glass-forming ability (GFA) of the alloy [17]. Kinetic reasons for improvement of GFA may be connected with the growth rate of the crystalline phases, stability of the supercooled liquid, etc. At the same time, devitrification process of multicomponent Ni–Nb–Ti–Zr–NM alloys is poorly studied.

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In the present work we study the effect of addition of a 4-d ETM Zr (substituting Nb) and 4-d late transition noble metals, namely Ag and Pd (substituting Ni) on devitrification of the  $\text{Ni}_{60}\text{Nb}_{25}\text{Ti}_{15}$  glassy alloy.

## 2. Experimental procedure

The ingots of nominal compositions  $\text{Ni}_{60}\text{Nb}_{25}\text{Ti}_{15}$ ,  $\text{Ni}_{60}\text{Nb}_{20}\text{Ti}_{15}\text{Zr}_5$ ,  $\text{Ni}_{55}\text{Pd}_5\text{Nb}_{20}\text{Ti}_{15}\text{Zr}_5$  and  $\text{Ni}_{55}\text{Ag}_5\text{Nb}_{20}\text{Ti}_{15}\text{Zr}_5$  alloys (at.%) were prepared by arc-melting mixtures of Ni 99.9, Nb 99.9, Zr 99.7, Ti 99.7, Pd 99.9 and Ag 99.9 mass% purities in an argon atmosphere. From these ingots, ribbon samples of about 20  $\mu\text{m}$  in thickness and 1 mm in width were prepared by rapid solidification of the melt on a single copper roller at a roller surface velocity of 42 m/s. The structure of the samples was examined by X-ray diffractometry with monochromatic Cu  $K\alpha$  radiation. The phase transformations were studied by differential scanning calorimetry (DSC) at a heating rate of 0.67 K/s and differential isothermal calorimetry. Transmission electron microscopy (TEM) investigation was carried out using a JEM 2010 (JEOL) microscope operating at 200 kV equipped with an energy dispersive X-ray (EDX) spectrometer of 0.1 keV resolution. The samples for TEM were prepared by the ion-polishing technique.

## 3. Results

TEM images in Fig. 1 as well as the XRD patterns indicate that the as-solidified alloys have an amorphous structure.

The DSC traces of the ribbon samples demonstrated in Fig. 2(a) show variation of  $C_p$  at the onset temperature of the glass transition ( $T_g$ ) marked with an arrow and the exothermic peaks (A, B and C) due to subsequent crystallization of the supercooled liquid. The addition of Zr and Pd increases the

supercooled liquid region of Ni–Nb–Ti alloy from about 45 K to about 50 K.

Compared to the ternary  $\text{Ni}_{60}\text{Nb}_{25}\text{Ti}_{15}$  glassy alloy which exhibits competing growth of oP8  $\text{Ni}_3\text{Nb}$  and cP2 TiNi phases upon crystallization [17] the addition of Zr and NM eliminates  $\text{Ni}_3\text{Nb}$  phase (Fig. 3). Even a small addition of Zr stabilizes oC68  $\text{Ni}_{10}(\text{Zr}, \text{Ti})_7$  phase which has been observed to form in Ni–Zr–Ti [13], Ni–Zr–Ti–NM [16] and Cu–Zr–Ti–Ni (in the form of  $(\text{Cu}, \text{Ni})_{10}(\text{Zr}, \text{Ti})_7$ ) [13] alloys. This phase follows precipitation of cP2 TiNi phase in  $\text{Ni}_{60}\text{Nb}_{20}\text{Ti}_{15}\text{Zr}_5$  alloy (Fig. 3). The lattice parameters of the  $\text{Ni}_{10}(\text{Zr}, \text{Ti}, \text{Nb})_7$  solid solution phase in  $\text{Ni}_{60}\text{Nb}_{20}\text{Ti}_{15}\text{Zr}_5$  alloy obtained by the least squares fitting are:  $a = 0.887$  nm,  $b = 0.885$  nm,  $c = 1.190$  nm, respectively. They are significantly lower than those of the binary  $\text{Ni}_{10}\text{Zr}_7$  phase due to dissolution of Nb and Ti substituting Zr.

The lattice parameter of cP2 TiNi phase in each studied alloy is somewhat smaller than  $a = 0.2972$  nm [18] of the binary cP2 TiNi phase without the solute elements. It is the smallest (0.293 nm) in the  $\text{Ni}_{60}\text{Nb}_{25}\text{Ti}_{15}$  alloy illustrating that Nb forms a solid solution substituting Ti. It is also confirmed by an energy dispersive X-ray analysis. However, the cP2 binary TiNi symbol will be farther used describing such a solution for simplicity. cP2 TiNi phase in  $\text{Ni}_{60}\text{Nb}_{20}\text{Ti}_{15}\text{Zr}_5$ ,  $\text{Ni}_{55}\text{Ag}_5\text{Nb}_{20}\text{Ti}_{15}\text{Zr}_5$  and  $\text{Ni}_{55}\text{Pd}_5\text{Nb}_{20}\text{Ti}_{15}\text{Zr}_5$  alloys has the lattice parameter of 0.294, 0.295 and 0.296 nm, respectively, as determined using (1 1 0) peak in Fig. 3. This also illustrates the effect of the alloying elements: Zr substitutes Ti and Nb while Ag or Pd substitute Ni increasing lattice parameter of cP2 TiNi as the dissolving atoms are larger in size than the dissolvent ones.

One can see that the diffraction peaks of cP2 TiNi phase in Fig. 3 are broadened. It allows to estimate the coherent scattering area size (crystallite size) using a procedure described in Ref. [19] and successfully used in various works, for example in Ref. [16]. The resulted coherent scattering area sizes

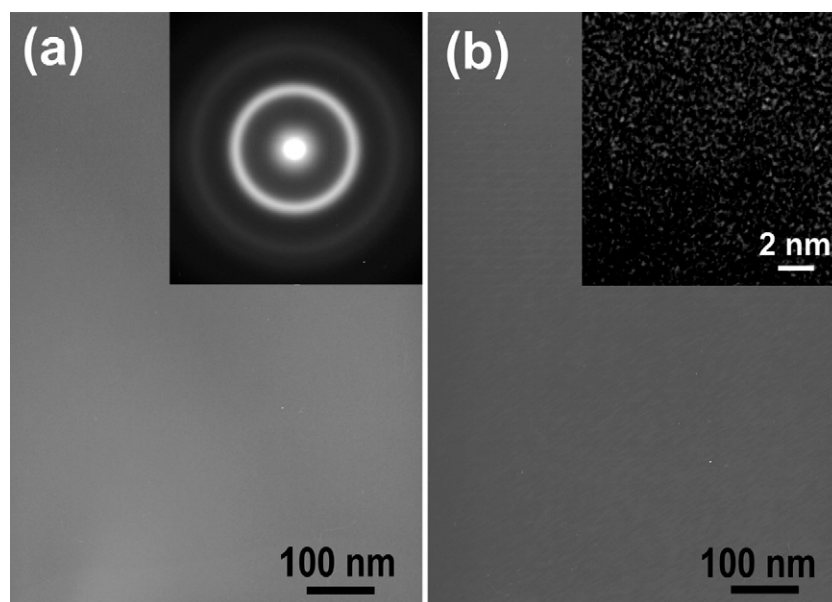


Fig. 1. Structure of  $\text{Ni}_{55}\text{Pd}_5\text{Nb}_{20}\text{Ti}_{15}\text{Zr}_5$  glassy alloy in as-solidified state, TEM. (a) Bright-field image, inset: selected-area electron diffraction pattern. (b) dark-field image, inset: high-resolution TEM image.

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