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# DSC, XRD and TEM characterization of glassy $Co_{40}Fe_{22}Ta_8B_{30}$ alloy with very high thermal stability

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#### ABSTRACT

In this letter we report about a new melt-spun  $Co_{40}Fe_{22}Ta_8B_{30}$  metallic glass with a wide supercooled liquid region of 74 K. X-ray diffraction (XRD) and high resolution transmission electron microscopy (HRTEM) investigations confirmed that crystallization occurs with significantly longer incubation time during annealing above the glass transition, as compared to the well-known  $Co_{43}Fe_{20}Ta_{5.5}B_{31.5}$  glassy alloy. The new alloy as a good soft magnetic material showed a very low coercivity of 0.8 A/m and polarization of 0.5 T. The origin of high thermal stability was attributed to the formation of Ta–Ta bonds in the microstructure and the absence or very low quantity of Ta–based units with the composition close to the stoichiometry of the (Co,Fe)<sub>21</sub>Ta<sub>2</sub>B<sub>6</sub> crystalline phase.

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#### 1. Introduction

Co-based bulk metallic glasses, BMGs, have attracted significant attention due to their high mechanical strength and good corrosion resistance as well as excellent soft magnetic properties [1,2].

In 2003, it was found that  $Co_{43}Fe_{20}Ta_{5.5}B_{31.5}$  glassy alloys exhibit ultrahigh fracture strength of 5 GPa and a wide supercooled liquid region of 70 K [1]. On the other hand, annealing above the glass transition temperature,  $T_g$ , results in the crystallization of  $(Co,Fe)_{21}Ta_2B_6$  phase with a very short incubation time, even when the annealing temperature is slightly above  $T_g$  [1]. BMGs with shapes and dimensions not accessible by solidification techniques could be produced by the consolidation of glassy powders above  $T_g$  [2]. In this relation, long incubation time prior to crystallization is essential for increasing the sintering time without the occurrence of crystallization. To our knowledge, there is no report about characterization and properties of Co-Fe-Ta-B glassy alloys with Ta content > 6 at% produced by rapid solidification. In this letter, we report a new glassy Co<sub>40</sub>Fe<sub>22</sub>Ta<sub>8</sub>B<sub>30</sub> alloy with a wide supercooled liquid region of 74 K and significantly longer incubation time produced by melt spinning.

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#### 2. Experimental procedure

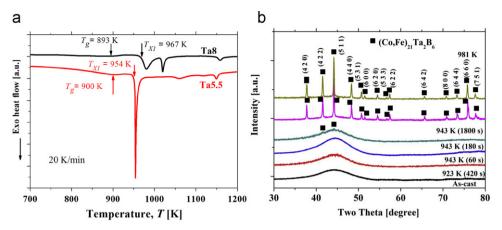
Master alloys with the nominal compositions  $Co_{40}Fe_{22}Ta_8B_{30}$  and  $Co_{43}Fe_{20}Ta_{5.5}B_{31.5}$  were prepared by arc-melting of pure metals (99.5 + %) under a Ti-gettered Ar atmosphere. The samples were re-melted several times to provide homogeneity; no weight-loss of the ingots has been observed. Amorphous ribbons were produced by single-roller melt spinning on a copper wheel under Ar flow. The glassy nature of ribbons was examined by X-ray diffraction in reflection geometry (XRD, Cu K $\alpha$  radiation), as well as in transmission geometry using a high energy monochromatic synchrotron radiation ( $\lambda$  = 0.123984 Å, BW5 beamline, HASYLAB at DESY Hamburg, Germany). The total structure factors, *S*(*Q*), where *Q* is the wave vector, were determined from the normalized elastically scattered intensity according to the Faber–Ziman formalism [3]. The total radial distribution function, RDF(*r*), was calculated by sine Fourier transform of *S*(*Q*) as described elsewhere [4].

The thermal stability and crystallization kinetics were investigated by differential scanning calorimetry (DSC, NETZSCH 404) under a flow of high-purity argon. The microstructure was characterized by high resolution transmission electron microscopy (HRTEM, Tecnai F30 operating at 300 kV).

#### 3. Results and discussion

Fig. 1(a) shows DSC curves of the as-cast  $Co_{40}Fe_{22}Ta_8B_{30}$  and  $Co_{43}Fe_{20}Ta_{5.5}B_{31.5}$  alloys measured at a constant heating rate of

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**Fig. 1.** DSC plots of as-quenched  $Co_{40}Fe_{22}Ta_8B_{30}$  and  $Co_{43}Fe_{20}Ta_{5.5}B_{31.5}$  glassy ribbons (a) and XRD patterns of  $Co_{40}Fe_{22}Ta_8B_{30}$  ribbon annealed above the glass transition at different times and temperatures (b).

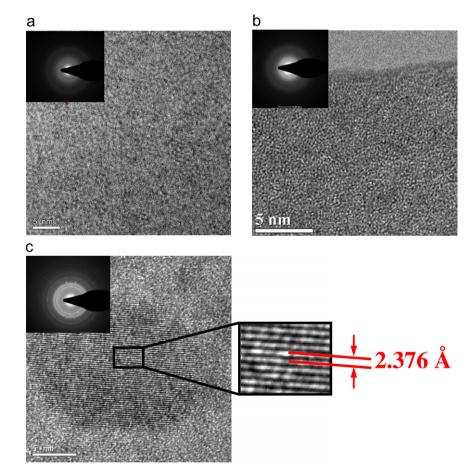


Fig. 2. HRTEM images and the corresponding SAED patterns for Co<sub>40</sub>Fe<sub>22</sub>Ta<sub>8</sub>B<sub>30</sub> glassy ribbon annealed for 420 s at 923 K (a), 60 s at 943 K (b) and 180 s at 943 K (c).

20 K/min (these alloys are called Ta8 and Ta5.5 hereafter in this work). The Ta8 alloy shows a clear glass transition with the onset at 893 K, followed by a supercooled liquid region  $\Delta T_X = 74$  K ( $\Delta T_X = T_x - T_g$ , where  $T_x$  is the crystallization temperature, measured at the onset of the first exothermic event) and crystallization at 967 K. In Ta8 alloy, the crystallization occurs through three exothermic peaks. On the other hand, in Ta5.5 alloy the onset of  $T_g$  is observed at 900 K and the primary crystallization occurs through a single exothermic reaction with a sharp peak at 953 K. Hence,  $\Delta T_x$  is 53 K in the Ta5.5 alloy, which indicates its lower thermal stability as compared to the Ta8 alloy. Measured  $T_g$ ,  $T_x$  and  $\Delta T_x$  for Ta5.5 alloy are lower than those reported in Ref. [1]

due to the lower heating rate in the DSC measurements (compare, 20 K/min in the present study and 40 K/min in Ref. [1]). It is well-known that the glass transition and crystallization temperatures shift to higher values upon increasing the heating rate.

Fig. 1(b) shows the XRD patterns of the as-quenched and annealed Ta8 alloy ribbons. The XRD plot of as-cast ribbon shows a broad halo pattern typical for fully glassy structure. The glassy state is preserved after annealing of Ta8 ribbons at 923 K ( $T_g$ +30 K) and 943 K ( $T_g$ +50 K) for 420 s and 60 s, respectively. In contrast, the crystallization of Ta5.5 alloy starts after annealing for 60 s at 928 K ( $T_g$ +18 K) [1]. Hence, the Ta8 alloy exhibits much longer incubation time prior crystallization even at a higher

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