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Full Paper

Study of the Characterization of Amorphous Alloy $Co_{87}Nb_{46}B_{15}$

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Abstract: This paper presents a discussion of the processing and characterization of alloy $Co_{87}Nb_{46}B_{15}$ produced by rapid solidification, in which characterization techniques such as X-Ray Diffraction (XRD), scanning electron microscopy and Spectroscopy Dispersion Energy (SEM / EDS) and magnetic properties were used, with the study of hysteresis curve. Several researches were done about nanocrystalline materials, especially cobalt-base alloys, because of their thermodynamic stability, characterization and processing. Amorphous alloys have a structure of ultrafine, homogeneous grains with small crystal clusters in amorphous phase.

Keywords: amorphous alloy $Co_{87}Nb_{46}B_{15}$; mechanical alloying; hysteresis curve

1. INTRODUCTION

The research in the field of metallic glasses started since the discovery of ability to amorphize $Au_{75}Si_{25}$ in 1960 by means by rapid solidification, the amorphous alloys and Bulk Metallic Glassy alloys (BMG) may be distinguished on the basis of cooling rates and the thermal response as the material is heated above room temperature [1].

The former needs very high cooling rate (~10^6 °C/s) to be amorphized while the latter can be achieved at lower quench rates. The nanocrystalline alloy may be obtained by controlled crystallization from amorphous precursors and was initially used as precursors to metallic glasses. These new amorphous alloys have high mechanical strength combined with good ductility besides allowing good plastic deformation in the region of supercooled liquid above the glass transition temperature, opening great technological prospects for processing amorphous materials.

The amorphous phase is of great technological importance, because of its applicability as a soft magnetic material, which has low cohesive strength, hysteresis loss and high permeability. Due to its versatility, the amorphous alloys have shown to have properties that can be used in various fields: such as, computer memories, core transformers for power electronics, reactors for coating, piping and electrodes for electrochemical processes.

The nanocrystalline grain structure is homogeneous and without segregations, these are the main features that give these alloys excellent magnetic properties combined with high resistance to corrosion. Amorphous materials can be produced by various methods, techniques: thermal evaporation (or vapor deposition) metals, sputtering (or sputtering), chemical vapor deposition or CVD (chemical vapor deposition), mechanical grinding high energy (mechanical alloying), melt spinning mechanical alloying, mechanical alloying and casting the electric arc furnace [2].

Amorphous metal alloys are obtained by rapid solidification from the liquid metal (molten) and have a disordered amorphous structure. The basic condition to obtain a material with extremely fine grains by crystallization can be attributed to the slow growth rate and nucleation, which is a characteristic of an amorphous solid. Thus, the cooling rate control in amorphous alloys will give result in a homogeneous distribution and the random nanocrystalline metal particles and precipitate in the amorphous matrix. The alloys of the binary Co-B system can be easily

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induced amorphization by melt spinning or high energy mechanical grinding. More complex alloys with specific properties can be obtained by adding other elements in this system, such as niobium, or gallium [3].

Amorphous alloys based on Co, Fe, Nb, B, and amorphous system of the type Co-Nb-B are easily fabricated using reaction technique of solid state and have good magnetic properties, thermal stability and a high saturation magnetization, high permeability, low coercivity and loss, which find their application in anti-theft security system, power electronics, telecommunications devices and automotive magnetism [4]. Similarly some alloys are based on amorphous alloys, and amorphous alloys of large volume (BMGs) as Co-Nb-B consisting of covalent bond formed by metalloid element (B) and a transition metal element with high cobalt modulus [5]. Moreover, niobium and boron have a negative enthalpy of mixing with the constituent in the majority Co.

The characterization of this amorphous alloy $Co_{87}Nb_{46}B_{15}$, obtained through the alloying process, was studied in this work. The characterization study was carried out by X-ray diffraction, scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) and magnetic measurements using vibrating sample magnetometry (MAV).

2. MATERIALS AND METHODS

The powders of the cobalt, niobium, and boron elements as its particle size (sieve per 100 mesh) have a purity of 99.99% each of the elements from Aldrich Chemical, were weighed in the appropriate proportions the composition ($Co_{87}Nb_{46}B_{15}$) and homogenized mechanically and the components were weighed on a precision scale, Micronal B4000 with 10^{-2}g resolution obtaining the nominal compositions.

The determined proportion ball / powder was 20:1, indicating the total sample weight, 25 g, but also the weight of the chrome steel ball, with three different sizes (6 balls of 20mm, 4 balls of 15 mm and 6 balls of 10 mm), for a total of ball 225g.

This material was placed in a hard steel jar, which was sealed to obtain vacuum 5.10^{-2} mbar, preventing possible contamination from powders. The jar with the mixture of elemental powders was placed in a planetary mill high energy, Model: NQM2L Mill Pulverizer which was initially crushed by 5h, rotation of 300 rpm.

A Shimadzu XRD 6000 diffractometer, a copper radiation ($CuK_{\alpha 1}$ = 1, 54056 Å) at 40 kV and 30 mA were used to identify the phases of the amorphous alloy ($Co_{87}Nb_{46}B_{15}$). The K_{β} contribution of the incident radiation is eliminated with a graphite monochromator.

The patterns were obtained in the continuous mode in the 2θ region between 10º and 80º, with a scan rate of $0.04s^{-1}$, and 0.02 º steps with acquisition time per channel 2.5s. The analysis by SEM/EDS was performed with a Shimadzu Superscan SSX-550 with an acceleration voltage of 0.5 to 30kV with a 10V step; afterwards the sample has been coated with a thin layer of gold deposited under vacuum in order to improve the image contrast.

Chemical analysis was carried through by energy dispersive spectroscopy (EDS) and the magnetic characteristic of the samples at low temperatures was carried out in PPMS (Quantum Design model MultiVu 6000 San Diego, USA).

3. RESULTS AND DISCUSSION

3.1 X-Ray Diffraction

The diffraction spectrum of the sample ray illustrated in Figure 1, without heat treatment, shows that there are no typical peaks of crystalline phases. It is only possible to notice a diffraction halo, evidencing the amorphous state in this sample, and confirming the vitreous aspect.

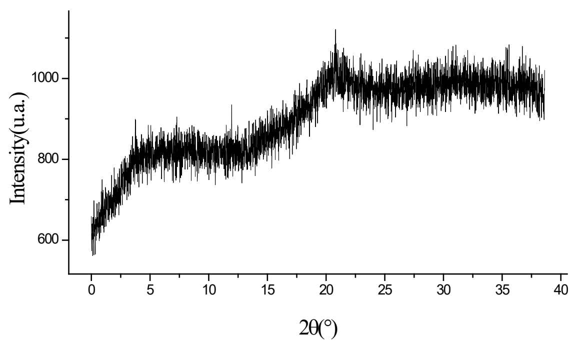

Figure 1. Spectrum of XRD amorphous alloy $Co_{87}Nb_{46}B_{15}$.

The simple expanded peak is characterized by totally amorphous structure, so we can say that the alloy has amorphous characteristics that prove successful amorphization of the $Co_{87}Nb_{46}B_{15}$ alloy studied with stoichiometry. It is clearly observed in the diffractogram there are some unidentified peaks and there is a central peak due to the presence of amorphous phases with the formation of a halo between 15º and 25º.

3.2 Scanning electron microscopy and EDS

Figure 2 shows respectively the amorphous alloy powder $Co_{87}Nb_{46}B_{15}$ the result of electron microscopy in the sample scan of the sample, showing an agglomerate of particles with irregular edges and small pores, where one can observe small amorphous flakes. This agglomeration is a common phenomenon resulting from the forces of Van der Waals. Moreover, fractures of the coarse and fine particulate cold welded coma occur simultaneously decreasing the fragility of collisions between the walls of the jar and the collision between the balls due to an increase of its temperature and kinetic energy. This led to the formation of smaller particles with irregular shapes and these agglomerates are 5µm size. The steady growth of oxide layer has been determined by the existing phases between cobalt and niobium in a given melting temperature 1200°C the formation of the alloy, thereby constant metaphases between amorphous and intermetallic phases are presented in grain with shapes of agglomerates of irregular and spherical shapes with few nodules on its entire surface.

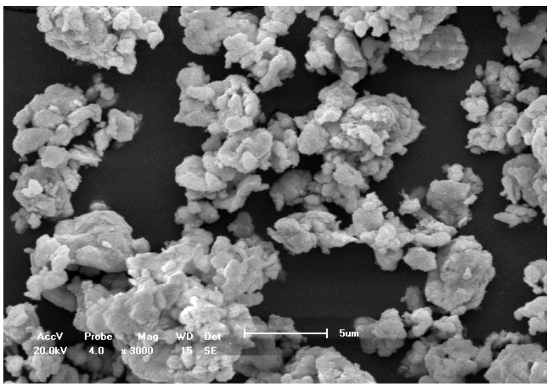

Figure 2. SEM $Co_{87}Nb_{46}B_{15}$ amorphous alloy forming irregular grains.

Figure 3 shows the EDS spectrum attached to SEM analyzing the presented elements in the particulate grinding 5 h $Co_{87}Nb_{46}B_{15}$ powder synthesis.

The dispersive energy of spectroscopy (EDS) is used in the analysis of amorphous alloy $Co_{87}Nb_{46}B_{15}$ with its respective spectrum covered with thin layer of gold. The cover of the sample is performed in a sputter apparatus in which is deposited a thin layer of gold or chromium 20 nm for 2 minutes at a voltage subjected of 5-6 mA under high vacuum.

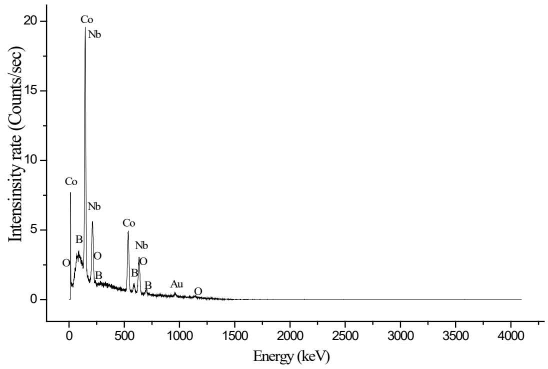

Figure 3. Analysis of EDS amorphous alloy $Co_{87}Nb_{46}B_{15}$.

After making the cover, the specimen may be fixed to the support using adhesive tapes of carbon. The cover and the mounting of the amorphous alloy sample aim to make it a good electrical conductor immobilize it in the holder and ensure that there is continuity between the sample and conductivity of the medium. This continuity is important to ground the specimen, avoiding the accumulation of charges on the sample. Additionally, for samples with higher atomic number, such as metals used for roofing, the depth of interaction volume is smaller but with higher production of secondary electrons resulting in images with higher contrast than those which were generated from samples of atoms with low atomic number, like most biological materials composed primarily of carbon, oxygen, hydrogen and nitrogen. The elements of higher concentration of the cobalt and niobium with small amounts of oxygen present peritectic reaction and allow the formation of oxides.

Boron is an amorphizator agent of amorphous structure. The intensities of these peaks in the EDS spectrum in a mentioned sample, where it is considered no loss in the percentage of atoms of this sample, being close to the real value of desired stoichiometry and giving around 100% of heavy components in analytical balance in phase the amount of pure metals, were placed in a high energy mill.

3.2 Magnetic properties

The measured magnetization was performed in a temperature range of 77-1000 K. The saturation magnetization values of 0K were obtained by extrapolation given saturation magnetization, as shown in Figure 4.

The magnetic anisotropy of the sample was determined by the sample rotation relative to a normal to the surface. The magnetic moment of the amorphous alloy behavior is compared with similar crystalline materials. The cooled sample shows a very smooth curve and a very sharp definition in the hysteresis curve near the surface that is rich in amorphous phase. The alloy has a high permeability accompanied by a small amount of coercive field of 0,008 Oe.

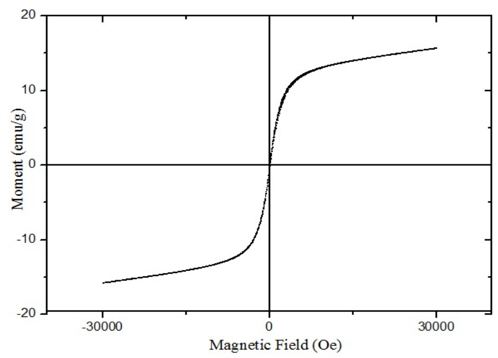

Figure 4. Hysteresis curve of amorphous alloy $Co_{87}Nb_{46}B_{15}$.

The presence of the metalloid boron, in this case, results in forming localized p-d type connections that reduce the number of polarizable-orbital rotation in the d orbital of the cobalt atoms and niobium [6]. Boron does form three covalent bonds using sp^2 hybrid orbitals in a trigonal plane. The transition from the polycrystalline to the amorphous phase is rather sharp: a large proportion of the alloy was in an amorphous phase state when boron concentration is intermediate within the atomic structure. The frameworks of such molecules may include atoms other than boron (e.g., carbon) and many of those with carbon (the carboranes) form complexes with transition metals. The lack of evidence for crystalline boride phases suggests that the B atoms occupy interstitial sites or measurements revealed a contraction of the lattice with increasing B content that the boride phase present are below the detection limit of the XRD.

Consequently, the coercivity was measured to decrease proportional to the increasing B content. The extended amplitude of peaks halos was reduced relative to both Co and Nb near neighbors due to the reduction in the chemical order and the introduction of static structural disorder by the presence of the B atom. Thus the rotation mostly has been linked directly below the Fermi level and boron atoms and niobium have a tendency to cluster around each other in these alloys, a result consistent with the large electronegativity difference and the greatest heat negative training between these two atomic types that create entangled state due to the emergence of various surface phenomena [7].

It is concluded about magnetic properties of amorphous alloy $Co_{87}Nb_{46}B_{15}$, that there is a difference in their magnetic moment because of a low property of ferromagnetic phases.

It should be emphasized that iron, cobalt and nickel are ferromagnetic materials. The ferromagnetism can be considered a special case of paramagnetism in which the magnetic moments of individual atoms align, and all point in the same direction. The magnetic susceptibility increases when this occurs compared with the situation where magnetic moments are not coupled. Many compounds of transition elements such as nanocrystalline alloys are paramagnetic material that contains partially-filled electron levels. The number of unpaired electrons can be calculated by measuring the magnetic moment.

The magnetochemistry of transition elements provides grants to state whether the d electrons are paired or not. These measures are of great importance to distinguish whether a given complex or octahedral oxide is high or low spin. The appearing of the magnetic properties of the amorphous alloy is due to the presence of its paramagnetism at room temperature with a strong antiferromagnetic coupling.

4. CONCLUSION

The expanded halo format with a simple peak is a characteristic of totally amorphous structure, so we can say that the alloy has amorphous characteristics. SEM analysis shows a mass of particles with distinct and well irregular morphology due to the process of amorphization and amorphous phases present in the amorphous alloy.

Depending on the initial mixture, structural change of mechanically milled powders occurs as follows: grain refinement, solid solution of diffusion and/or the formation of new amorphous phases where we can see the EDS largest cobalt concentrations, niobium and boron formed oxides with oxygen.

It is concluded about magnetic properties of amorphous alloy $Co_{87}Nb_{46}B_{15}$, that there is a difference in their magnetic moment because of a low property of ferromagnetic phases.

The appearing of the magnetic properties of the amorphous alloy is due to the presence of its paramagnetism at room temperature with a strong antiferromagnetic coupling.

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5. ACKNOWLEDMENTS

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