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Amorphous nickel nanophases inducing ferromagnetism in equiatomic Ni–Ti alloy

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ABSTRACT

$Ni_{50}Ti_{50}$ nm-sized amorphous particles are prepared using inert-gas condensation followed by in situ compaction. Elemental segregation of Ni and Ti is observed in the consolidated nanostructured material. Amorphous, nearly pure Nickel (96%) nanophases form within the amorphous $Ni_{50}Ti_{50}$ alloy. Combining atom probe tomography and scanning transmission electron microscopy with computer modelling, we explore the formation process of such amorphous nanophase structure. It is shown that the Ni rich amorphous phase in the consolidated nanostructured material is responsible for the ferromagnetic behavior of the sample whereas the rapidly quenched amorphous and crystalline samples with the same chemical composition ($Ni_{50}Ti_{50}$) were found to be paramagnetic. Due to the high cooling rate obtained using the inert gas condensation technique, an exceptional control over the crystallization processes is possible, promoting the formation of various amorphous phases, which are not obtained by standard rapid quenching techniques. Our findings demonstrate the potential of amorphous metallic nanostructures as advanced technological materials, and useful magnetic compounds.
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1. Introduction

In many aspects modern technologies are focused on crystalline materials because of their beneficial magnetic [1], electrical [2], mechanical [3], and chemical [4] properties. However, the recent advances in experimental and modelling methods have opened new areas of investigation in the field of amorphous solids [5,6].

In this regard, a new family of non-crystalline solids termed as “nanoglasses” has been proposed by Gleiter and co-workers [6], which are fabricated by consolidating quenched nanometer-sized amorphous or glassy particles obtained with either thermal evaporation [7] or magnetron sputtering [8] in an inert gas atmosphere. The consolidated nanosized particles consist of amorphous regions, which are connected by interfaces characterized by a lower density and higher free volume compared to the amorphous nanoparticle cores [6]. Molecular dynamics (MD) simulations have shown that these interfaces are characterized by a short- and medium-range order and an excess free volume different from the bulk [5,9]. Evidence to support the existence of such interfaces in consolidated nanostructured materials has been gathered from the combination of small angle X-ray diffraction and positron annihilation lifetime spectroscopy [10]. The findings have been further confirmed by Mössbauer spectroscopy (MS) investigations of Fe-Sc nanoglasses, which clearly show interfacial regions that are a few atomic layers in thickness with a reduced density and a larger distribution of distances between nearest neighbor atoms [11]. This structural model is further supported by the fact that the volume fraction of the interfaces was found to vary inversely with the size of the amorphous nanoparticles and confirmed by observing the isomer shift in Mössbauer spectra of Pd–Fe–Si alloy [12]. Although the structural studies have confirmed the high free volume in the interfacial regions, it is not clear whether the free volume is a

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chemical or structural effect. Recently, Cu and Fe enriched regions have been observed in $Cu_{50}Zr_{50}$ and $Fe_{90}Sc_{10}$ systems, respectively [13,14]. By comparing $Pd_{80}Si_{20}$ and $Cu_{64}Zr_{36}$ amorphous nanoparticles, Adjaoud et al. [15] demonstrated that palladium segregates to the shell and silicon segregates to the core in the Pd–Si system, while copper migrates to the shell and zirconium to the core in Cu–Zr nanospheres. As a result of such novel nano-amorphous structure, new properties of non-crystalline materials can be expected. In fact, they exhibit spectacular properties, such as high catalytic activity [16], improved mechanical performance [10], and enhanced magnetic characteristics [11], which differ significantly from any known amorphous material. Specifically, it has been shown by means of micro-compaction and tensile tests that such consolidated nanostructured materials have excellent plastic deformation ability compared to the melt spun ribbon with identical chemical composition. Such an improvement in ductility is probably due to less dense interfaces, which accommodate deformation thereby avoiding the fatal nucleation of system-spanning shear bands. A change in magnetic properties of the consolidated nanometer-sized amorphous particles in comparison to the reference ribbon has been observed in $Fe_{90}Sc_{10}$ alloy [11,17]. It has been demonstrated that the contribution of the itinerant electrons to the magnetization is different in nano-amorphous structure because of the high free volume in the interfaces.

In this regard, we synthesized amorphous nanoparticles of equiatomic Nickel-Titanium (Ni–Ti) similarly by inert gas condensation and thoroughly characterized their structural and magnetic properties. $Ni_{50}Ti_{50}$ alloys are in widespread use in robotic, engineering, medical, and aerospace applications [18–20], due to their favorable corrosion resistance, high hardness, and good electrical conductivity. While the alloys show unique properties as shape memory alloys and for bio-compatibility properties, to date, no ferromagnetic state has been reported for this material. In this study, we report on the finding of ferromagnetism in $Ni_{50}Ti_{50}$ consolidated nanostructured materials, and trace this to the effect of the interfacial regions. These are found to consist of amorphous Nickel phases with distinct ferromagnetic properties differing from those of the conventional paramagnetic crystalline and amorphous materials with the same chemical composition.

2. Methods

$Ni_{50}Ti_{50}$ amorphous nanoparticles were prepared by using magnetron sputtering in inert gas condensation system (IGC), which has been described elsewhere [14]. The main difference to conventional thin film sputtering process is the much higher inert gas pressure in the chamber on the order of 10^{-1} mbar. Pellets with a diameter of ~8 mm, and a thickness of ~300 μm were then fabricated by *in-situ* compaction at a uniaxial pressure of 3 GPa, followed by *ex-situ* compaction in a high pressure torsion machine at 6 GPa. Such consolidated structure will be referred to as the amorphous nanophase materials (ANM's). The reference sample, amorphous metallic thin film (AMTF), was synthesized by Direct Current magnetron sputtering at a working pressure of 8×10^{-3} mbar. The base pressure of the magnetron sputtering chamber was ~10^{-8} mbar. The thickness of the films was ~900 nm. In order to avoid contamination with oxygen, a 5 nm Gold (Au) capping layer was deposited *in-situ* on top of the Ni–Ti thin film. Crystallization of the thin film samples was initiated by annealing in an ultra-high vacuum furnace with a residual gas pressure of 1×10^{-8} mbar at 650 °C for 60 min. The compositions of the ANM and AMTF were measured using an energy dispersive X-ray spectroscopy (EDX) (Oxford Instruments X-MaxN 50 mm^2 Silicon Drift Detector) in a LEO 1530 scanning electron microscope. The structural properties of the samples were determined by X-ray diffraction (XRD) using a Philips and Bruker X-ray diffractometer equipped with a Mo-Kα and Cu-Kα radiation sources, respectively. Transmission electron microscopy (TEM) was carried out using FEI Tecnai F20-ST and FEI Titan 80–300 aberration corrected microscopes. The powder sample used for TEM analysis was collected directly from the cold finger in the IGC chamber using a glass test tube, which was transferred to the microscope in a special vacuum holder to prevent exposing the sample to air. Preparation of TEM lamellae and tips for atom probe tomography (APT) samples was performed by employing both FEI Strata 400 and Zeiss Auriga 60 focused ion beam systems (FIB). Prior to the lift-out, a platinum protection layer (150 nm) was deposited over the area of interest to protect the sample surface from gallium ion beam milling damage and sample degradation. To produce the required atom probe specimen geometry, annular milling was used to create needle-shaped morphology with a tip diameter smaller than 100 nm. More details about the FIB milling process can be found in Ref. [21]. The APT measurements were carried out using a Cameca-LEAP 4000× HR instrument in laser pulse mode (wavelength 355 nm, pulse frequency 100 kHz, pulse energy 60 pJ, evaporation rate 0.50%) at 50 K. Data processing was achieved with the CAMECA integrated visualization and analysis software (IVAS-version 3.6.1), incorporating standard reconstruction algorithms, allowing the extraction of three-dimensional nanoscale chemical distribution of all detected atoms in the analysis volume. The magnetic properties were investigated using the Quantum Design MPMS 5XL (SQUID) magnetometer.

MD simulations were performed using the large-scale atomic/molecular massively parallel simulator code [22]. A modified embedded-atom method potential was used to describe the interatomic interactions in the Ni–Ti system [23]. The chosen potential gives a good fit to basic properties of pure crystalline Ni, pure crystalline Ti, and Ni–Ti alloys. To replicate the synthesis procedure in IGC, we prepared the formation of nanoparticles by repeatedly adding 100 atoms (50 Ni atoms and 50 Ti atoms) with velocity on the order of kilometer per second in a virtual reaction chamber. The system was then equilibrated at 800 K for 100 ps. This process was repeated until a total of 20000 atoms were added to the chamber. XRD line profiles from the simulations were obtained by using the virtual diffraction algorithm [24].

3. Results and discussion

The nature of the amorphous nature of ANM and AMTF is confirmed by X-ray diffraction and Transmission electron microscopy (see Fig. 1(a–e)). A completely crystallized AMTF after annealing at 650 °C for 1 h is also shown in Fig. 1(a). XRD peak profile shows only the B2 phase without any other phases. In addition, X-ray diffraction profiles were calculated based in the atomistic simulations. The corresponding profiles show a good match to the experimental ones, which will be discussed later. Fig. 1(b) shows a representative TEM image of the as prepared powder with sizes varying between 3 and 15 nm. The non-crystalline nature of the prepared nanoparticles is confirmed by the selected area electron diffraction (SAED) in Fig. 1(c), which is composed of broad diffraction halos without any crystalline diffraction spots. All these observations clearly indicate that the nanometer sized particles are completely amorphous. After high pressure compaction, the amorphous structure is still retained as can be seen in the XRD and high resolution TEM images as well as the electron diffraction patterns (see Fig. 1(a) and (d)). The elemental distribution mapping of the ANM sample obtained from the scanning transmission electron microscopy using an EDX shows Ni-rich and Ti-rich regions separated from each other (see Fig. 1(f)) on the length scale of about 10 nm. No segregation was observed in

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