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Preparation and Magneto-Structural Investigation of High Ordered (*L2₁* Structure) Co2MnGe Microwires

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Preparation and Magneto-Structural Investigation of High Ordered (*L2₁* Structure) Co_2MnGe Microwires

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Abstract: We used the Taylor-Ulitovsky Technique to prepare nanocrystalline Co_2MnGe Heusler alloy glass-coated microwires with a metallic nucleus diameter of 18 ± 0.1 µm and a total diameter of 27.2 ± 0.1 µm. Magnetic and structural studies were carried out to determine the fundamental magneto-structural characteristics of Co_2MnGe glass-coated microwires. XRD revealed a well-defined nanocrystalline structure with average grain size about 63 nm, lattice parameter a = 5.62 and a unique mixture of $L2_1$ and B2 phases. The magnetization curves for field cooling and field heating (FC-FH) demonstrate a considerable dependence on the applied magnetic field, ranging from 50 Oe to 20 kOe. Internal stresses, originated by the production process, resulted in various magnetic phases, which were responsible for the notable difference of field cooling (FC) and field heating (FH) curves on magnetization dependence versus temperature. Furthermore, the ferromagnetic behavior and expected high Curie temperature together with high degree of $L2_1$ ordered makes it a promising candidate for many applications.

Keywords: microwires; Heusler alloys; Magneto-structural characterization; Secondary phases; $L2_1$ & B2 phases structure

1. Introduction

Heusler alloys, discovered at the beginning of 20-th century, are a diverse family of binary, ternary, and quaternary compounds with a wide range of physical characteristics suitable for various applications, including spintronics, magnetic refrigeration, actuators among others. [1,2]. They pique the curiosity of fundamental and applied researchers due to their considerable tunability depending on chemical composition, crystal structure, or electrical structure [3]. Spin polarization, superconductivity, shape memory, and magnetocaloric effect, in particular, have attracted considerable interest from both an experimental and theoretical point of view [1-3]. Heusler alloys, also known as full-Heusler alloys with the stoichiometry X_2YZ, may be classified into many classes based on their chemical composition and consequent characteristics [4]. Heusler based on Co_2YZ is a prominent category of materials with high spin polarization (P) or even half metallicity (P 100%). However, theoretical and experimental investigations show that spin polarization is very sensitive to structural instability. The $L2_1$ crystalline phase has the greatest structural ordering, which is necessary to achieve the requisite spin polarization levels. While the mutual exchange of atoms on the Y-Z position (B2 disorder) has little effect on spin polarization values, the X-Y or X-Y-Z disorders (D03 or A2, respectively) can dramatically reduce spin polarization [5]. Moreover, Heusler alloys described above, have complex crystalline structures that need extremely high temperatures (usually > 1000 K in the bulk form and > 650 K in the thin-film form) for their crystalline ordering [5]. As a result, one major issue when producing X_2YZ full-Heusler thin films is to achieve the chemically-ordered $L2_1$ phase as the excellent features of Co_2-based Heusler compounds (Co_2MnGe) are most typically

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expected for this $L2_1$ phase (see Figure 1a). Co_2-based Heusler alloys, on the other hand, can crystallize in a variety of phases with reduced chemical ordering without affecting the atomic sites in the lattice [7]. The most common disordered phase is B2, in which Y and Z atoms are randomly distributed, resulting in a primitive unit cell rather than the FCC cell (Fm-3m → Pm-3m) (see Figure 1b). At this time, it is unknown how much the chemical disproportion affects the physical features. It's worth noting that, *ab initio* calculations [8] and experiments ones [9] show that the physical properties (Curie temperature, cell parameter, magnetic moment, magnetic damping constant, and spin polarization at EF) of the $L2_1$ and B2 phases are slightly distinguishable from one another, and the half-metallic spin gap should be conserved.

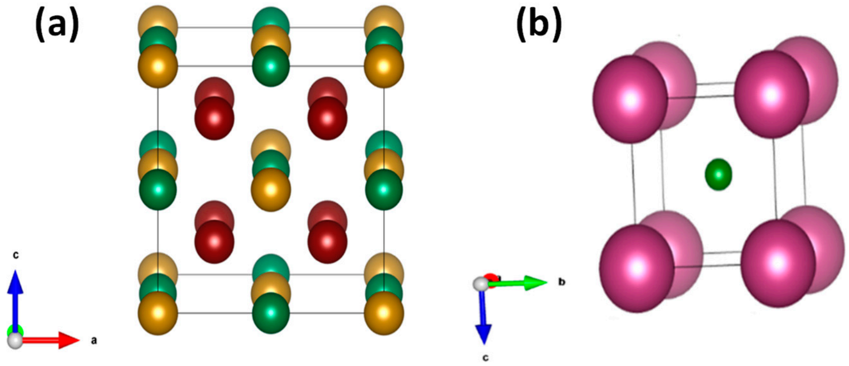

Figure 1. (a) L21 cubic structure (Co atoms occupy the red positions, Mn atoms occupy the yellow positions and Ge atoms occupy the green positions) and (b) B2 cubic structure (Co atoms occupy the purple positions, Mn and Ge atoms occupy the green positions).

In the current work, we present an attempt to obtain Co_2MnGe glass-coated microwires. The fabrication method choice is due to the interesting magneto-structural behavior of glass-coated microwires from Heusler alloys as well as functional properties of glass-coated microwires, such as superior mechanical properties, insulating, thin and flexible glass-coating and thin dimensionality [10-19]. Accordingly, we manufactured Co_2MnGe glass-coated microwires using the Taylor-Ulitovsky process described in details elsewhere [12,13]. The Taylor-Ulitovsky technique, known since the 1960s [20], is one of the fabrications recently used to produce Heusler alloys glass-coated microwires [10-12, 14-19]. The fundamental advantage of this low-cost technology is that it enables the manufacturing of thin and long (a few kilometers long) microwires with an extended diameters range (d- values from 0.1 to 100 μm) at high speeds (up to a few hundred meters per minute) [20-22]. Glass-coated microwires with outstanding mechanical characteristics are also produced using this technology [13,23-25]. The glass coating on the microwires can provide us additional benefits, such as increased insulation and environmental protection. Furthermore, the availability of a biocompatible thin, flexible, insulating, and highly transparent glass covering might help biological applications [26,27]. As a result, Heusler microwires based on Co_2MnGe are a potentially smart material for a wide variety of devices applications. To our best knowledge, no one has reported on preparation, and structurally, mechanically, or magnetically characterization of Co_2MnGe-based glass-covered Heusler microwires.

2. Materials and Methods

For the production of Co_2MnGe glass-coated microwires, the first step is the manufacturing of the Co_2MnGe alloy ingot by arc melting under argon atmosphere. The melting process starts by melting the nominal elements with high purity (Co (99.99%), Mn (99.9%) and Ge (99.9%)). The argon atmosphere is proceeded, in order to avoid the oxidation during melting. To attain an alloy with higher homogeneity the melting route was repeated five times. Then, the nominal composition was verified by performing Energy Dispersive X-ray (EDX) analysis, finding the real composition to be

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$Co_{55}Mn_{22}Ge_{23}$. Afterwards, when we acquire the alloy, we prepare the Co_2MnGe glass-coated microwires through the Taylor-Ulitovsky technique [12,17,20]. This fabrication procedure consists of drawing and forming directly from the melted master alloy, then additional chemical composition has been performed as illustrated at Table 1. The obtained diameter of inner metallic nucleus of microwire sample is around 18 μm, while the total diameter (with an external Pyrex coating) is around 27.1 μm. The microstructure and phase composition analysis for the produced samples have been examined with a BRUKER X-ray diffractometer (D8 Advance, Bruker AXS GmbH, Karlsruhe, Germany), performed with Cu Kα (λ = 1.54 Å) radiation. Furthermore, the magnetic behavior was scrutinized through the magnetization curves, which were measured using a PPMS (Physical Property Magnetic System, Quantum Design Inc., San Diego, CA) vibrating-sample magnetometer at temperatures, T, between 5 and 400 K. A magnetic field, H, from 50 Oe to 20 kOe was applied along the sample axis and perpendicular to the wire axis. The results are provided in terms of the normalized magnetization, M/M_{5K}, where M_{5K} is the magnetic moment obtained at 5 K.

Table 1. Atomic percentage of Co, Mn and Ge elemental composition in Co_2MnGe glass-coated microwires.

EDX spectrum	Co (at. %)	Mn (at. %)	Ge (at. %)
Average	56	19	25

3. Results

To check the chemical composition of Co_2MnGe glass-coated microwires we performed EDX/SEM analysis and the output results listed in Table 1 The composition of the metallic nucleus was found to be somewhat different from the stoichiometric one using the EDX data from Table 1 (Co_2MnGe). This little variance was due to the peculiarities of the preparation procedure, which included alloy melting and casting. We examined the nominal composition for 10 locations to determine the amount of difference. The actual 2:1:1 ratio for Co, Mn and Ge was verified for all locations, with an atomic average $Co_{56}Mn_{19}Ge_{25}$.

X-ray diffraction (XRD) patterns of Co_2MnGe alloys in glass coated microwires are investigated at room temperature and shown in Figure 2. As shown in Fig.2, at low angles, the XRD diffractogram, at $2\theta \approx 22^{\circ}$ a huge halo is observed, which must be ascribed to the amorphous glass coating presence. The same behaviour was reported and discussed elsewhere, [10-19]. The Co_2MnGe full Heusler alloy has to be indexed in the Fm-3m space group with an $L2_1$ cubic structure. Indeed, the cubic structure of Co_2MnGe is confirmed from the XRD profile. From the XRD pattern analysis, it is well perceived the presence of a cubic structure. Nevertheless, a second phase can be detected (see Figure 2b). This hypothesis is drawn from the fact that the main peak (220) results from an overlapping of two very close peaks; one peak at around $2\theta \approx 44^{\circ}$, recognized to the $L2_1$ structure and the other one at about $2\theta \approx 45^{\circ}$, which may be due to the B2 type disordered structure.

Therefore, the crystalline structure of prepared Co_2MnGe glass-coated microwires is an FCC-$L2_1$ with a minor BCC-B2 cubic structure in some parts of the synthesized sample. Although the $L2_1$ structure is an extremely ordered and crucial structure to achieve the required spin polarization values, but it should be mentioned that the manufacturing process of the microwires may results a different structural disorder (B2, A2, DO3, etc.) as described in the introduction. For instance, the mutual disorder between Y (Mn) and Z (Ge) atomic positions outlines the B2 disorder type (see Figure 1). It is well noting that, the theoretical calculations anticipated that the B2 type disorder structure produced in Y-Z elements has a far smaller impact on the spin polarization values than the X-Y disorder and X-Y-Z disorder, both of which noticeably diminish this feature [5]. The presence of (111) and (200) and (311) super lattice diffraction peaks confirm the presence of high ordered of $L2_1$ structure [28]. The estimated lattice parameter and calculated volume of the cell are a = 5.7430 Å and V = 189.42 Å^3, respectively, which perfectly matched with the lattice parameter and Volume values reported elsewhere [9, 29] for Co_2MnGe thin films. Such of high ordered $L2_1$ structure is for the first time detected in Co_2MnGe-based glass-coated microwires.

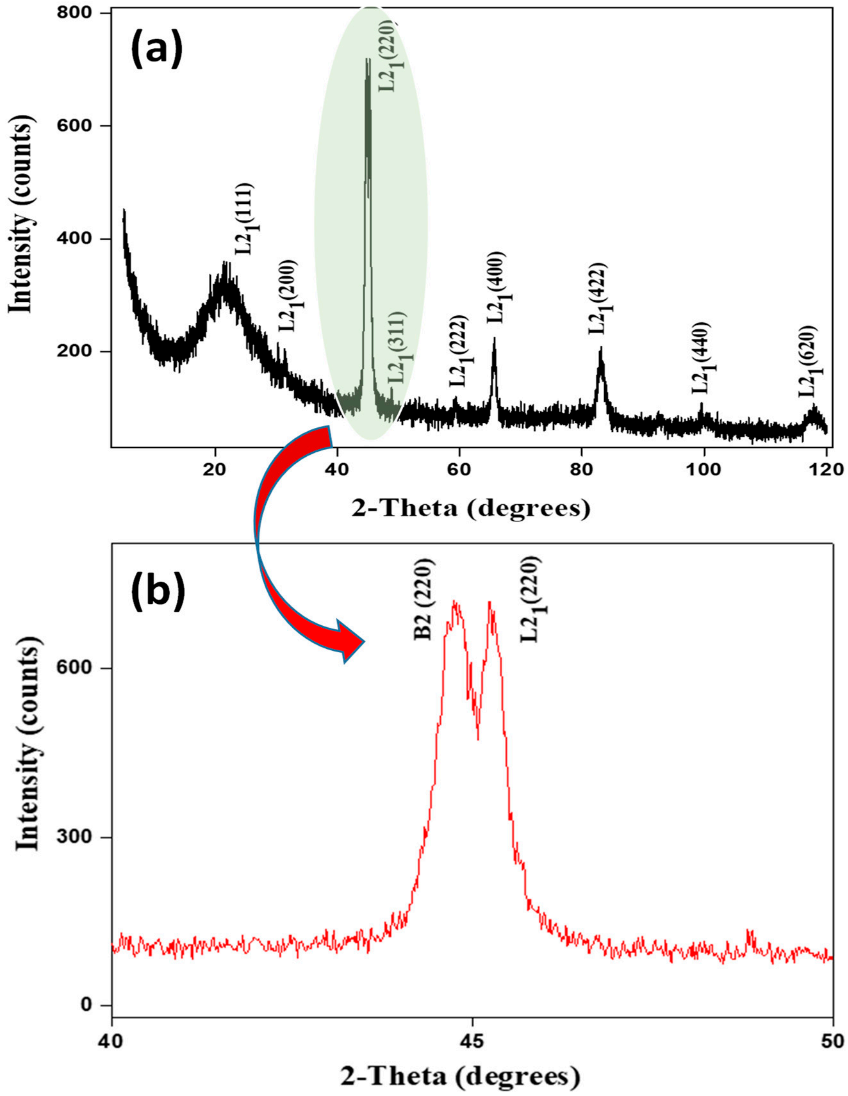

Figure 2. (a) X-ray diffraction (XRD) diffractograms at room temperature of Co_2MnGe and (b) is XRD diffraction patterns of Co_2MnGe (enlargement of 2 2 0 peaks).

For deeper investigation of the microstructure of Co_2MnGe we used the Debye- Scherrer's equation, described in our previous works [16]. Using this protocol, we can estimate the average grain size, D_g, related to each peak, being for as-prepared Co_2MnGe microwires of about 63.3 nm.

The ferromagnetic ordering of as-prepared Co_2MnGe glass-coated microwires is evidenced from Figure 3, where the magnetic hysteresis (M-H) loops measured at $5K \leq T \leq 305$ K are provided. The M-H loops have been measured at applied high magnetic field, H, up to ±40 kOe to make sure that Co_2MnGe glass-coated microwires sample present magnetic saturation. In addition, M-H loops were measured at different temperature to illustrate their behavior with temperature. Due to the high ordered $L2_1$ structure perfect ferromagnetic behavior is observed where the normalized saturation magnetization has a monotonic increase by decreasing the temperature i.e., the lowest value of M/M_{5K} ratio detected at 305 K and the highest value is observed at 5K. Thus, the high degree of $L2_1$ ordered phase of as-prepared as well as the average grain size, D_g, of Co_2MnGe glass-coated microwires are relevant factors, that can affect M-H behavior with temperature. Thus, such character of M-H loops by varying the temperature was not observed in our previous investigation for Co_2Mn-based glass-coating microwires due to the low degree of $L2_1$ ordered phase (see [12,17]).

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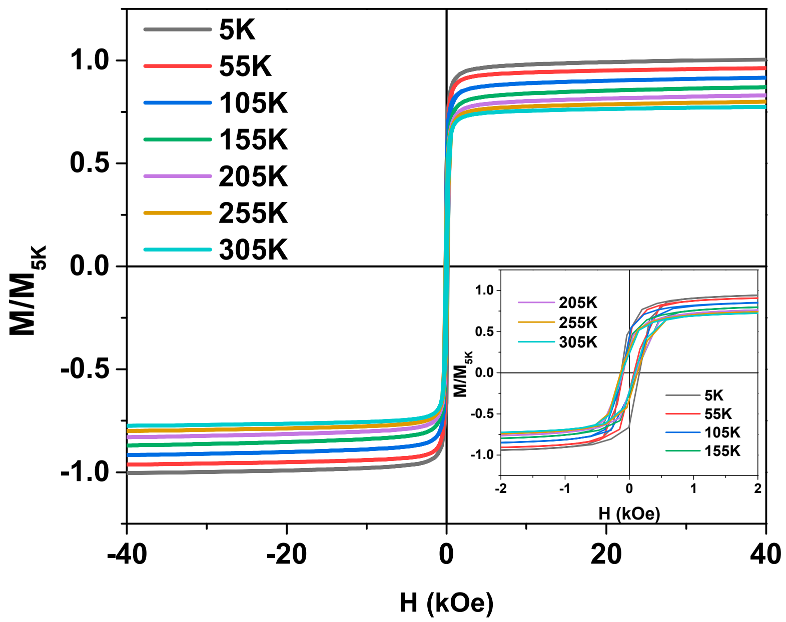

Figure 3. Magnetization curves M/M_{5K} (H) of as-prepared Co_2MnGe glass-coated microwires measured at maximum field ±40 kOe and temperature range 305 K to 5K. Low field M/M_{5K} (H) loops are shown in the inset.

From low field hysteresis loops (see inset of Figure3) the coercivity, H_c, of about 120 Oe for whole T range can be appreciated. Such H_c –values are about one order of magnitude higher than that reported for other Co_2Mn –based microwires [12,17]. This difference in H_c –values can be related to higher average D_g –values observed in as-prepared Co_2MnGe glass-coated microwires.

The thermomagnetic properties i.e., (M/M_{5K}) vs. T and magnetic field of Co_2MnGe glass-coated microwires are shown in Figure 4 and Figure 5. In this part, we only focused on the magnetization behavior at a wide range of temperature and magnetic field to evaluate the possible magnetic phase transition. We measured temperature dependencies of the magnetization in the temperature, T, range from 5 to 400 K. To avoid the over estimation of the magnetization we used the normalize magnetization parameters (M/M_{5K}), where the M_{5K} is the highest magnetic moment detected at 5K. A notable ferromagnetic behavior has observed for all range of measuring temperature and applied magnetic field, which is expected due to the high Curie temperature for Co_2MnGe alloy above 883 K [9,29]. For field cooling (FC) and field heating (FH) magnetization curves a notable mismatching between FC and FH curves have observed when (M/M_{5K} vs T) dependence measured at low magnetic field i.e., 50 Oe and 200 Oe as seen in Figure 4. For the M/M_{5K} (T) curves measured at 50 Oe the FC curve overlap FH curves for temperature range 400 K to 200 K, then reversed for temperature range 200 K to 30 K and finally full matching is observed for T below 30 K. This behavior can be discussed with two flipped points where the FC and FH magnetization curves changed. By increasing the external applied magnetic field i.e., 200 Oe, these flipping points are disappeared and uniform magnetic tendency is detected, where FH overlaps FC for the temperature range 400 K to 20 K, while perfectly matching below 20 K, as indicated in Figure 4b.

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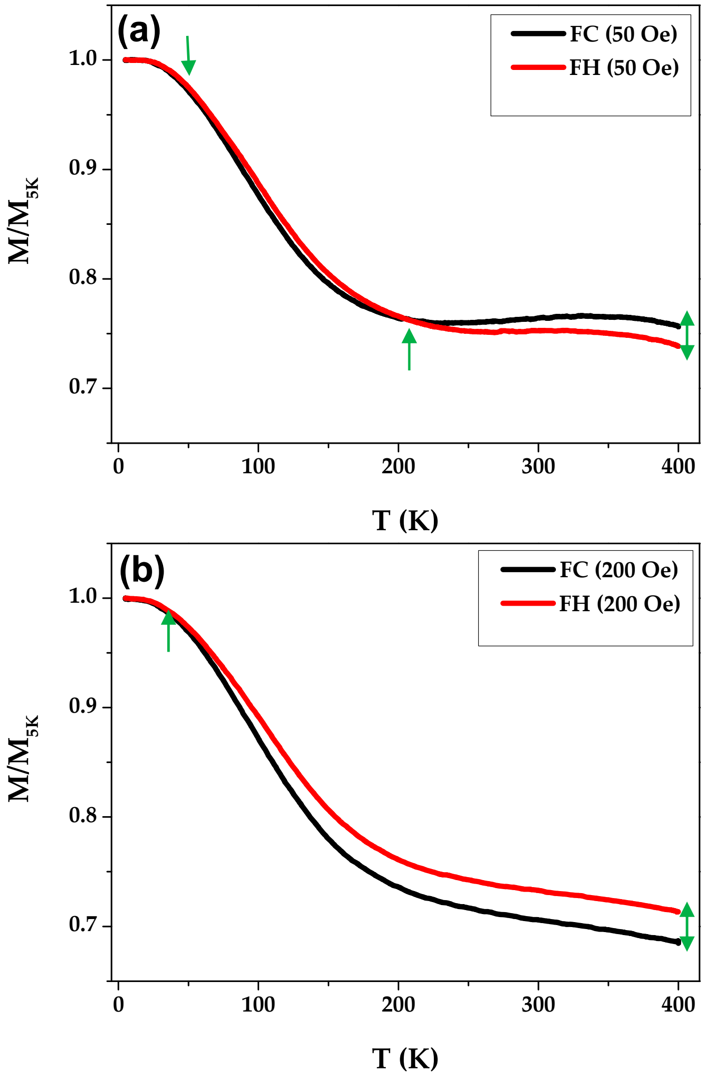

Figure 4. Measured temperature dependence of magnetization for as-prepared Co_2MnGe glass-coated microwires with 50 Oe and 200 Oe of applied external magnetic field.

For further increase of the applied magnetic field, both of FC and FH magnetization curves are perfectly matched and homogenous ferromagnetic behavior is seen (see Figure 5). The interesting magnetic field dependence of FC and FH curves indicates the sensitivity of Co_2MnGe to the magnetic field and temperature.

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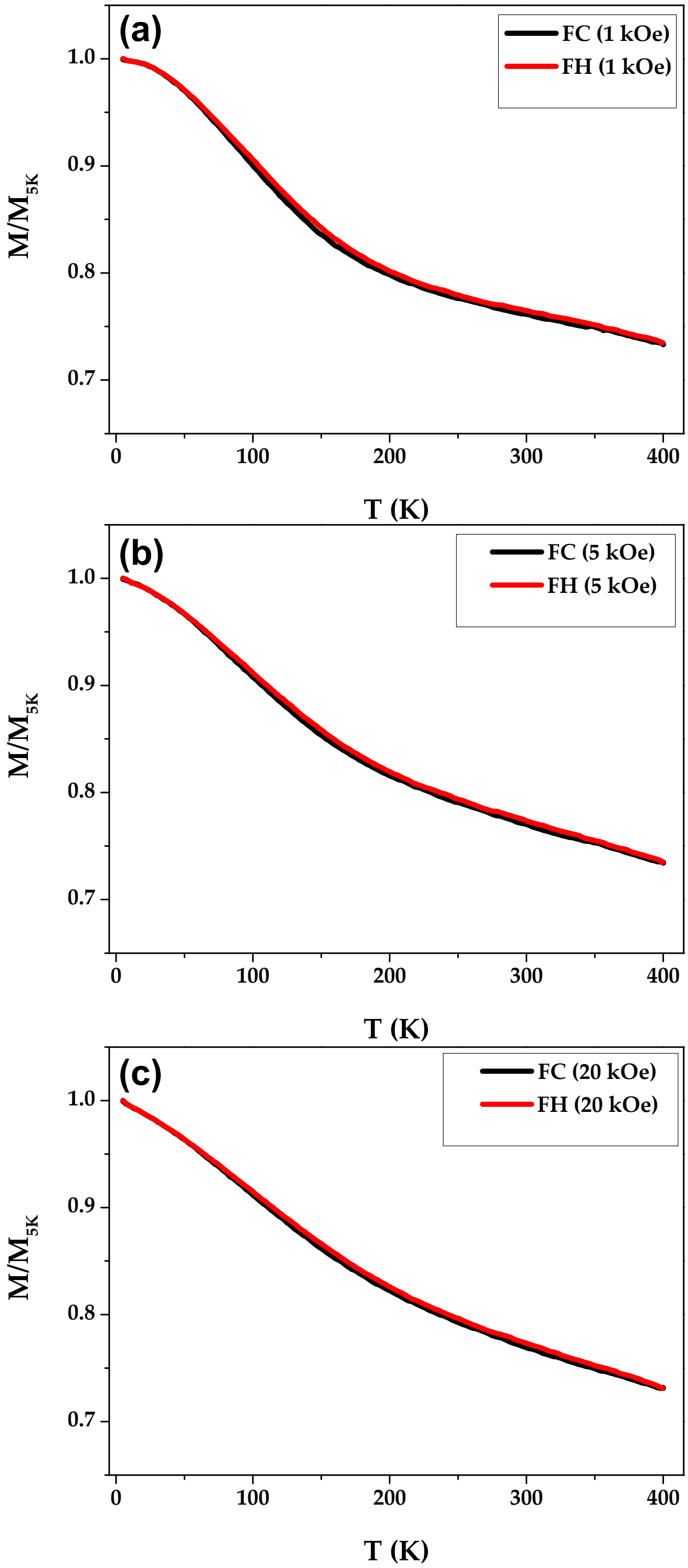

Figure 5. Measured temperature dependence of magnetization for as-prepared Co_2MnGe glass-coated microwires with 1 kOe, 5 kOe and 20 kOe of applied external magnetic field.

4. Discussion

The strong variation of the FC and FH magnetization curves with external applied magnetics field must be related to the microstructure of Co_2MnGe-glass-coated microwires and with the peculiarities of the fabrication method. As illustrated in XRD analysis the high ordered $L2_1$

microstructure is confirmed beside to the disordered B2 phase structure. The existence of B2 disorder phase structure strongly affect the magnetic behavior at applied low magnetic field i.e., 50 and 200 Oe, resulted the two flipped point and the mismatching between the FC and FH magnetization curves. This disordered effect is totally canceled by applied high magnetic field 1 kOe (in our case) to 20 kOe. Thus, a perfect matching of FC and FH magnetization curves observed (see Figure 5). The increase of degree of microstructure ordering of Heusler-based glass coated microwires leads to a uniform magnetic behavior with temperature and magnetic field for more details see [11, 16-19]. The origin of such disordered structure must be related to rapid melt quenching involved in the fabrication method [15,19, 30-32]. Alongside the disordered structure, the preparation of glass-coated microwires is also characterized by large internal stresses (up to 1 GPa), originated mainly by the essentially different thermal expansion coefficients of metallic alloy and glass-coating [30-32]. On the other hand, such disordered structures has been also observe in thin films [33]. It is worth mentioning that the structural disorder and high internal stresses in glass-coated Heusler alloy microwires and thin films can be considerably diminished by appropriate annealing [33,34]. Therefore, one of the future line of research of Co_2MnGe glass coated microwires will be search for the appropriate postprocessing for magnetic properties tunning.

5. Conclusions

In summary, we report on fabrication of a high ordered Co_2MnGe glass coated microwires by using Taylor-Ulitovsky technique. In as-prepared Co_2MnGe microwires ferromagnetic ordering is observed in the whole range of temperatures. The XRD analysis confirm the present of high ordered nanocrystalline $L2_1$ structure with average of crystallite size 63.3 nm and with lattice constant of 5.7430. Besides to the $L2_1$ structure, a disordered B2 structure is found combing with the main peak of $L2_1$. The existence of B2 disordered structure can explain the mismatching of FC and FH magnetization curves. By increasing the external applied field, the effect of disordered B2 microstructure is totally suppressed and uniform magnetic behaviour is seen for applied magnetic field higher than 1 kOe. Future investigations are needed due to study the effect of high ordered microstructure on different physical properties. The out coming result reveals the promising Co_2MnGe with high spin polarized and $L2_1$ ordered structure in multifunctional thermomagnetic application.

Author Contributions: Conceptualization, M.S. and A.Z.; methodology, V.Z. M.I.; validation, M.S., V.Z. and A.Z.; formal analysis, M.S and A.W.; investigation, M.S., A.W, and A.Z.; resources, V.Z. and A.Z.; data curation M.S, M.I. and A.W; writing—original draft preparation, M.S., A.W. and A.Z.; writing—review and editing, M.S. and A.Z.; visualization, M.S., A.W., and V.Z supervision, A.Z.; project administration, V.Z. and A.Z.; funding acquisition, V.Z., and A.Z. All authors have read and agreed to the published version of the manuscript.

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