

A new high-toughness Co-based nanocrystalline soft-magnetic alloy capable of large stress-induced anisotropy

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Abstract:

Mechanical and magnetic properties for a new Co-base nanocrystalline soft magnetic alloy, $\text{Co}_{80}\text{Fe}_{1.5}\text{Mn}_{1.5}\text{Nb}_8\text{Si}_2\text{B}_7$ at.%, are compared to those of previously studied $\text{Co}_{(80-2x)}\text{-Fe}_x\text{-Mn}_x\text{-Nb}_4\text{-Si}_2\text{-B}_{14}$ at.% (where $x = 2.3, 2.7$, and 4) as cast and stress-annealed ribbons. The new alloy exhibits a fracture toughness, K_{IC} , of $58 \text{ MPa}\sqrt{\text{m}}$ after stress-annealing at 500°C and a stress-induced/stress-normalized magnetic anisotropy, K_u/σ , of $0.094 \text{ kJ}/(\text{MPa}\cdot\text{m}^3)$, i.e., a value nearly double that of other Co-base nanocrystalline alloys. The bulk magnetostriction, λ_s^{bulk} , of the new composition was also determined to be approximately zero after stress-annealing. XRD was used to identify the nanocrystalline phases in annealed ribbon, and the fracture morphologies were observed via SEM. The fracture surfaces of tough and brittle annealed ribbons contained morphologies indicative of local melting and traditional shear hackle/mist patterns, respectively, suggesting that the iron and manganese concentrations in Co-based nanocomposites influence the alloy's mechanical properties and processability.

Since the 1980's, nanocrystalline soft magnetic materials (NSMM), such as FINEMET, have offered improved performance for many electromagnetic components including tape-wound transformers and inductors [1]. When FINEMET ribbon is wound into a toroid and used as the core of an inductor, it can exhibit lower losses and higher maximum operating induction ($B_s > 1$ T) than pressed-powder ferrite and gapped cores at intermediate frequencies (1 kHz–200 kHz). Moreover, it is capable of exhibiting tunable magnetic anisotropy, K_u , which is achieved through subsequent processing techniques including stress-annealing (SA) and/or field-annealing [2-6]. Despite certain obstacles, continuous in-line stress-annealing of NSMM can result in excellent soft-magnetic properties suitable for common and differential mode chokes, i.e., those that require high B_s and low relative permeabilities (μ) which prevent magnetic saturation at high peak currents. Specifically, FINEMET alloys can reach $\mu \sim 200$ by the application of ~ 200 MPa tensile stress during annealing [4, 5, 7]. The use of Fe-based NSMM in low μ applications, however, is hindered by the inherent brittleness that accompanies its nanostructure after annealing. The nanocrystalline composite structure, achieved by annealing a glassy precursor above a crystallization temperature, is necessary to achieve the maximal B_s , balance magnetostriction, and activate the anisotropy mechanism(s) that tune K_u and μ [8-10]. Prior works have shown that a maximum K_u of ~ 15 kJ/m³ was attainable in narrow short stress-annealed strips of Fe-based ribbon with stresses up to 800 MPa [11]. But there is no record of successfully SA wider ribbon in large quantities at comparable stress levels due to its poor mechanical properties. This observation is consistent with other works that show these alloys exhibit low mechanical toughness ($K_C \approx 2$ MPa $\cdot\sqrt{m}$) [7, 12-14].

More recently, a family of cobalt (Co)-based NSMM have been developed [11, 15-19] that can achieve low μ with SA and are suitable for high current applications including differential-mode chokes. Interestingly, this family of NSMM is not as susceptible to annealing-induced embrittlement as its Fe-based counterparts. Co-based NSMM have demonstrated a slightly lower B_s of ~ 1 T [20], but a stress-induced K_u [2, 11, 15, 16, 21] three times larger, thus enabling $\mu < 200$. Moreover, Co-based NSMM are known to exhibit a higher Curie temperature, T_c , than related Fe-based systems [22], yielding higher temperature stability and near-zero magnetostriction [22, 23]. Typically, these alloys include Co, Fe, Mn, Nb, Si, and B and their relative concentrations influence B_s , T_c and K_u [20, 22, 24-27]. Crystallites that nucleate within the amorphous precursor during annealing tend to be close-packed structures [16, 26, 27]. Work by Leary et al. has suggested that the source of stress-induced K_u in Co-based NSMM stems from planar defects in these close-packed phases [11], which are sensitive to composition-dependent virtual bound states (VBS) [11, 16]. Hence, Fe and Mn were added in small quantities to influence K_u -stress sensitivity and balance magnetostriction while retaining a high B_s [27-29]. Some of the most promising NSMM in this family are $\text{Co}_{(80-2x)}\text{-Fe}_x\text{-Mn}_x\text{-Nb}_4\text{-Si}_2\text{-B}_{14}$ (at.%) where $x = 2.3 - 4$. In alloys with $x = 2.7$ and 4, a K_u of 10.8 kJ/m³ [2]

and 14 kJ/m³ [11], respectively, can be achieved by SA at only 200 MPa, resulting in a $\mu \sim 30$ in an alloy with $B_s = 1$ T. However, only few studies report on the improved mechanical properties of these promising new alloys.

One recent paper on Co–Fe_{2.7}Mn_{2.7}Nb₄Si₂B₁₄ at.% found that the ultimate tensile strength decreased from 2289 MPa to 1453 MPa in as-cast and crystallized ribbon, respectively [30]. The ribbon's elastic modulus increased from 165 GPa to 190 GPa with crystallization, while Poisson's ratio remained nearly constant at 0.3. The modulus and Poisson's ratio has been related to fracture toughness, K_C , in prior works pertaining to Fe-based NSMM [12, 13], whereby a decrease in Poisson's ratio and bulk modulus were observed due to annealing-induced relaxation and crystallization processes. Since these newer Co-based alloys are expected to exhibit the same deformation mechanism of other nanocomposites, i.e., shear banding [31, 32], it is expected that they too exhibit embrittlement with annealing. Unfortunately, the K_C has not been widely reported for Co-based NSMM, even though it is fundamental in defining their processability.

In this letter, we present K_C for the more commonly studied Co_(80-2x)-Fe_x-Mn_x-Nb₄-Si₂-B₁₄ at.% alloys where $x = 2.3, 2.7$, and 4 [2, 3, 11, 16-19, 21, 26, 27, 30], then compare the magnetic and mechanical properties of strips to the new composition, i.e., Co₈₀Fe_{1.5}Mn_{1.5}Nb₈Si₂B₇ at.%. The new alloy exhibits 25% higher K_C after SA and a K_u approximately two times as sensitive to mechanical stress, thus reducing the stress levels necessary to achieve low μ and minimize ribbon fractures during the SA process. These improvements were made without any decrement in B_s following the chemical substitution, demonstrating that the new alloy is a potential candidate for high current differential-mode chokes. Finally, bulk magnetostriction, λ_s^{bulk} , was found to be near-zero in the new alloy, which is a promising indication that minimal core losses can be reached with future processing optimization.

At least 600 m-long by 2.57 cm-wide continuous spools of amorphous precursor Co_(80-2x)-Fe_x-Mn_x-Nb₄-Si₂-B₁₄ ($x = 2.3, 2.7$, and 4) and Co₈₀Fe_{1.5}Mn_{1.5}Nb₈Si₂B₇ (Nb8) ribbon were cast from individual melt spin runs using a pilot-scale planar-flow melt spin system. The $x = 2.3$ (2.3), 2.7 (2.7), 4 (4), and Nb8 ribbons exhibited average thicknesses of $17.2 \pm 0.94 \mu\text{m}$, $19.5 \pm 0.95 \mu\text{m}$, $19.7 \pm 1 \mu\text{m}$, and $20.4 \pm 1 \mu\text{m}$ along their lengths, respectively, measured in increments of 30 meters along the ribbons' length. Differential scanning calorimetry indicated primary crystallization took place around 425°C in all of the alloys. Magnetic properties were measured in strips using a Laboratorio Elettrofisico strip tester at 400 Hz per the standard method [33]. The as-cast ribbon was processed using a custom in-line SA system [4, 5] which applied tensile stresses, σ , up to 500 MPa to ribbon fed through a furnace with a 4 second annealing time at 500 °C. The K_C was quantified by loading stress-annealed specimens with edge-cracks in tension to mode-1 failure while the load was recorded. Specimens were intentionally cracked by slicing the exposed edge of a ribbon sandwiched between two rubber-coated steel plates with a razor edge. The K_C was approximated as $K_C \cong$

$\sigma_f \beta \sqrt{\pi a}$ [7] where σ_f was the maximum nominal tensile stress of pre-cracked ribbon at failure, β was a geometry factor which depends on sample width (see [7] [34]), and a was the original edge crack length. Fracture morphology was observed via scanning electron microscopy (SEM). X-ray diffraction was performed in transmission-mode while exposing the airside of the stress-annealed ribbons to a molybdenum source. The baseline-corrected stress-induced K_u was computed for the Nb8 ribbon from linear BH loops as $K_u = B_s^2 / (2\mu\mu_0) - K_{u,0}$, where B_s is saturation induction in Tesla, μ is the relative permeability at an induction of 0.8 T, μ_0 is the permeability of free space ($4\pi \times 10^{-7} \text{ H/m}$), and $K_{u,0}$ the anisotropy (measured as $B_s^2 / (2\mu\mu_0)$) in the 0 MPa annealed condition. The intrinsic anisotropy present in Nb8 as-cast amorphous precursor ribbon was also measured to determine the effect of annealing with zero-stress. λ_s^{bulk} was indirectly measured in strips that exhibited linear BH loops by applying a tensile load between tests and was computed as $\lambda_s^{bulk} = -2\Delta K_u / (3\sigma_\lambda)$, where σ_λ was the applied tensile stress during the measurement, and ΔK_u was the change in magnetic anisotropy energy from the applied σ_λ [4, 5, 7, 35].

Figure 1(a) depicts the BH loops of the as cast, annealed only (0 MPa), and the stress-annealed Nb8 ribbon processed at 500 °C for 4 seconds under several σ levels. Due to the limitations of the strip testing device, the 20 μm – thick strip would only reach B_s if small annealing stresses were used. For the as-cast, 0 MPa, and 45 MPa SA-only condition, the ribbon saturated at approx. 1 T. Anisotropy fields for the three conditions were 0.86 kA/m, 2.4 kA/m and 10 kA/m, respectively, while H_c was 59 A/m, 315 A/m and 293 A/m for each condition. Moreover, the strips exhibited a remanence of 0.14 T, 0.1 T and 0.04 T, and permeabilities of 1037, 346 and 79.6, respectively. Conversely, when 400 MPa was applied, saturation was not observed and the ribbon exhibited ~ 750 A/m coercivity, 0.01 T remanence, and $\mu = 10$. Assuming saturation remained unchanged with the higher stress condition, the σ -induced baseline-corrected K_u in this alloy was found to be 38.6 kJ/m³ from a σ of only 400 MPa. As-cast precursor ribbon exhibited a $K_{u,0}$ of 0.383 kJ/m³, while the ribbon annealed at 500°C for 4 seconds under zero stress exhibited a $K_{u,0}$ of 1.15 kJ/m³.

As a comparison with the prior Co-rich NSMM, Figure 1(b) depicts linear BH loops for the Nb8 and x = 2.3, 2.7, and 4 ribbons following the SA process at 500 °C – 300 MPa for 4 seconds. The plot clearly shows that the Nb8 alloy exhibited a higher stress-induced K_u for the same processing σ . Figure 1(c) and (d) show the μ – σ and K_u/σ – σ relationships for these alloys. In Figure 1(d), the K_u/σ of x = 2.3, 2.7, and 4 were not corrected with respect to any baseline and were all approximately 0.045 kJ/(MPa·m³). Two K_u/σ – σ curves are depicted in the figure for the Nb8 ribbon, where the dashed line for “Nb8*” denotes the behavior corrected with respect to the as-cast precursor baseline of 0.383 kJ/m³, and the symbols for Nb8 represents the K_u/σ – σ relationship corrected with respect to the 1.15 kJ/m³ baseline from zero-stress annealed ribbon. By comparing the two Nb8 curves, it was evident that devitrifying the Nb8 ribbon under

zero uniaxial stress at 500°C for 4 seconds caused an increase in K_u by 0.767 kJ/m³. The K_u/σ appeared to plateau around 0.094 kJ/(MPa·m³) in the Nb8 ribbon for both baseline-corrected datasets when ~200 MPa uniaxial stress was applied, i.e., more than double that of the other ribbon compositions.

This type of data is not only useful for identifying sources of anisotropy, but it can also be employed for determining the appropriate SA parameters for choke core design [2]. For example, it is often beneficial for the μ of a core to be “functionally graded” so that the inner core radius exhibits a lower μ than the outer radius [2, 3]. Such a graded μ core forces even distribution of magnetic flux across the cross-section, i.e., preventing flux crowding. If the inner radius experiences flux crowding, cores will exhibit higher losses and local saturation, rendering a portion of the overall core useless. Minimizing flux crowding through μ grading is useful for both lightweighting and minimizing losses during high-duty cycle operation.

As shown by the data in Figures 1(c) and (d), if a core required a μ of 20 at the inner radius, the Nb8 ribbon would need ~220 MPa of tensile stress applied during annealing at 500°C and the other alloys would require ~420 MPa, or 113 N and 226 N, respectively for ribbon of similar cross section. Depending on the presence of small edge cracks (or casting defects), applying a high stress to reach lower μ could cause repeated ribbon breaks defeating the ability to manufacture radially-graded μ cores. Figure 1(d) depicts the stress-normalized K_u - σ plot clearly detailing the Nb8 alloy’s capability of minimizing the necessary processing stress over a wide range compared to the other Co-base alloys.

In addition to the higher K_u - σ sensitivity, the Nb8 alloy exhibited an enhanced K_C , even after SA above the alloy’s glass relaxation temperature. The average K_C (over 10 tests) was plotted in Figure 2(a) for the x =2.3, 2.7, and 4 alloys and Nb8 composition as a function of Fe + Mn concentration in at.%. The data was measured from ribbon strips (see Figure 1a) subjected to a 500 °C – 200 MPa – 4 second SA treatment, which reduced the ribbon’s mechanical strength compared to the as-cast amorphous state [30]. Interestingly, K_C trended almost linearly with Fe + Mn concentration after SA and the Nb8 strips (with Fe_{1.5} and Mn_{1.5}) exhibited the highest average K_C of 57.8 MPa· $\sqrt{m}$, even after SA. As stated earlier, Fe and Mn were added to influence K_u - σ , B_s , and λ_s^{bulk} . Prior work found that Mn concentration can effect chemical partitioning in these alloys [26, 27], but the effect on mechanical properties is not yet clear.

Figure 2(b) depicts the B_s and λ_s^{bulk} in the present alloys as a function of Fe + Mn concentration measured in strips subjected to the same processing treatment as those in Figure 2(a). As shown by the trend in B_s , nearly 1 T was reached in the x =2.7 and x =4 alloys, while the measured B_s in x =2.3 had decreased slightly to 0.96 T, but this value was within the expected standard error of the mean of the equipment. Interestingly, further decreasing the Mn and Fe concentration and increasing the Nb concentration from 4 at.% to 8 at.% resulted in a B_s = 1 T. This was higher than expected since the trend

from previous results for Co-based alloys showed a decrease in B_s with increasing Nb concentration [22, 25].

As shown in Figure 2(b), the measured λ_s^{bulk} followed a consistent trend, increasing as the Fe+Mn concentration increased, with the Nb8 alloy having a value closest to zero after SA at 500 °C and 200 MPa. Several works have reported a small negative λ_s^{bulk} for Co-enriched amorphous phases [21-23]. Upon annealing the amorphous ribbon, phases nucleate and grow which can affect magnetic properties including λ_s^{bulk} . In the present Nb8 composition, we found coexisting FCC and BCC phases via room-temperature X-ray diffraction in contrast to the faulted close-packed phases in the Fe/Mn~2 alloys (see Figure 3). The spectra were offset from the top to the bottom of the figure in ascending K_c order to discern differences in structure that may govern their mechanical properties. The BCC phase appeared to increase with increasing Fe+Mn content, having the most prominent peaks in the Nb =4 alloy, which corresponded to a minimum K_c . The inset depicts spectra for each composition between a 2θ of 28 and 44 for clarity. All the ribbon compositions were found to contain the same cubic phases. The Nb8 composition, however, exhibited a larger FCC lattice parameter than the other alloys and high enough FCC counts around $\approx 20^\circ$, so the peak was observed through the amorphous peak resulting in a visual split. The Nb8 ribbon with $K_c = 57.8 \text{ MPa}\cdot\sqrt{m}$ also appeared to contain BCC phases like the x =4 composition with a K_c of only $12.1 \text{ MPa}\cdot\sqrt{m}$. But the x =4 composition did not exhibit high intensity FCC peaks when compared to the other ribbons. The weak unlabeled peak at $\approx 18.5^\circ$ in the Nb8 ribbon was found to be from the (422) reflection of a cubic 23:6 phase, or surface oxide [36] (lattice parameter $a \approx 1.059 \text{ \AA}$) and was believed to be partially responsible for the larger H_c observed in the BH loops of Figure 1a. Peaks labeled as “*FCC” were those corresponding to close-packed structures (HCP/FCC) that were not impacted by stacking order, i.e., faulting, which may be one of the governing mechanisms of stress-induced K_u in these alloys [11, 16]. The data in Figure 3 demonstrates that the microstructure in Co-rich NSMM can influence both magnetic and mechanical properties, but the actual microstructural-property relationships are a matter of ongoing study. Further processing optimization and microstructural work is needed to fully understand the interactions between these nanostructures and their resulting behaviors in Co-based NSMM.

Finally, Figures 4(a)-(c) are examples of the fracture morphologies of the x =2.3, 2.7, and 4 ribbons from the K_c test coupons used to determine the fracture data shown in Figure 2(a). The x =2.3, 2.7, and 4 ribbons exhibited an average K_c of 45.1, 33.9, and $12.1 \text{ MPa}\cdot\sqrt{m}$ respectively, with distinctly different fracture topographies. On the fractured x =2.3 ribbon (see Figure 2(a)), triple vein points and a Taylor’s fluid meniscus were seen. This topography has been previously observed in glassy ribbons that exhibited local melting at shear bands from adiabatic loading [37, 38]. A 20-point average indicated a vein spacing of $\approx 0.764 \mu\text{m}$ with a $0.246 \mu\text{m}$ standard deviation. Wallner lines [39] were also observed along the sample

edge evenly spaced by $\approx 5 \mu m$ and indicate the direction of crack propagation. The fractured $x = 2.7$ ribbon (Figure 2(b)) did not exhibit Taylor fluid meniscus or vein topology, but rather shear hackle [39] with an average ridge spacing of $1.35 \mu m$ indicating a more brittle failure surface. The $x = 4$ ribbon, on the other hand, exhibited both shear hackle and “mist” [39] morphologies, indicating that it was the most brittle of the alloys consistent with the lowest K_C . Local shear hackle was only observed near the surface of the ribbon extending $1.7 \mu m$ into the cross section. Shear hackle peaks in the $x = 4$ sample were separated by an average of $0.42 \mu m$. In Figure 2(d) the Nb8 fracture morphology is shown, closely resembling the $x = 2.3$ ribbon with the higher K_C .

Given these observations, the Fe and Mn in these Co-based NSMM may be responsible for the density of shear transformation zones, the presence of atomic free volume, or cavitation ahead of the propagating crack tip [32], which generally serve as shear band nucleation sites [31, 37]. Shear banding is currently the most widely recognized deformation mode of NSMM [31, 40]. Apparently, the addition of Nb (up to 8 at.%) does not negatively impact the deformation mechanism governing toughness in the present nanocomposites and concurrently serves to maintain a high B_S , increase $K_u - \sigma$ sensitivity, decrease λ_s^{bulk} , and improve SA processability.

In this letter, the K_C , $K_u - \sigma$ sensitivity, B_S , and λ_s^{bulk} were quantified for Co-Fe_{2.3}-Mn_{2.3}-Nb₄-Si₂-B₁₄, Co-Fe_{2.7}-Mn_{2.7}-Nb₄-Si₂-B₁₄, Co-Fe₄-Mn₄-Nb₄-Si₂-B₁₄, and Co-Fe_{1.5}-Mn_{1.5}-Nb₈-Si₂-B₁₄ at.% (Nb8) alloys. The Nb8 alloy, though comprised of the same constituents, exhibited 25% higher K_C after in-line SA and a twofold $K_u - \sigma$ sensitivity leading to the ability to manufacture high-current gapless differential mode chokes with $B_S = 1$ T and a stress-induced K_u as high as ~ 40 kJ/m³. λ_s^{bulk} was found to be nearly zero, which was proportional to the Fe+Mn concentration. These findings may offer pathways to improved processability in NSMM and future choke core optimization.

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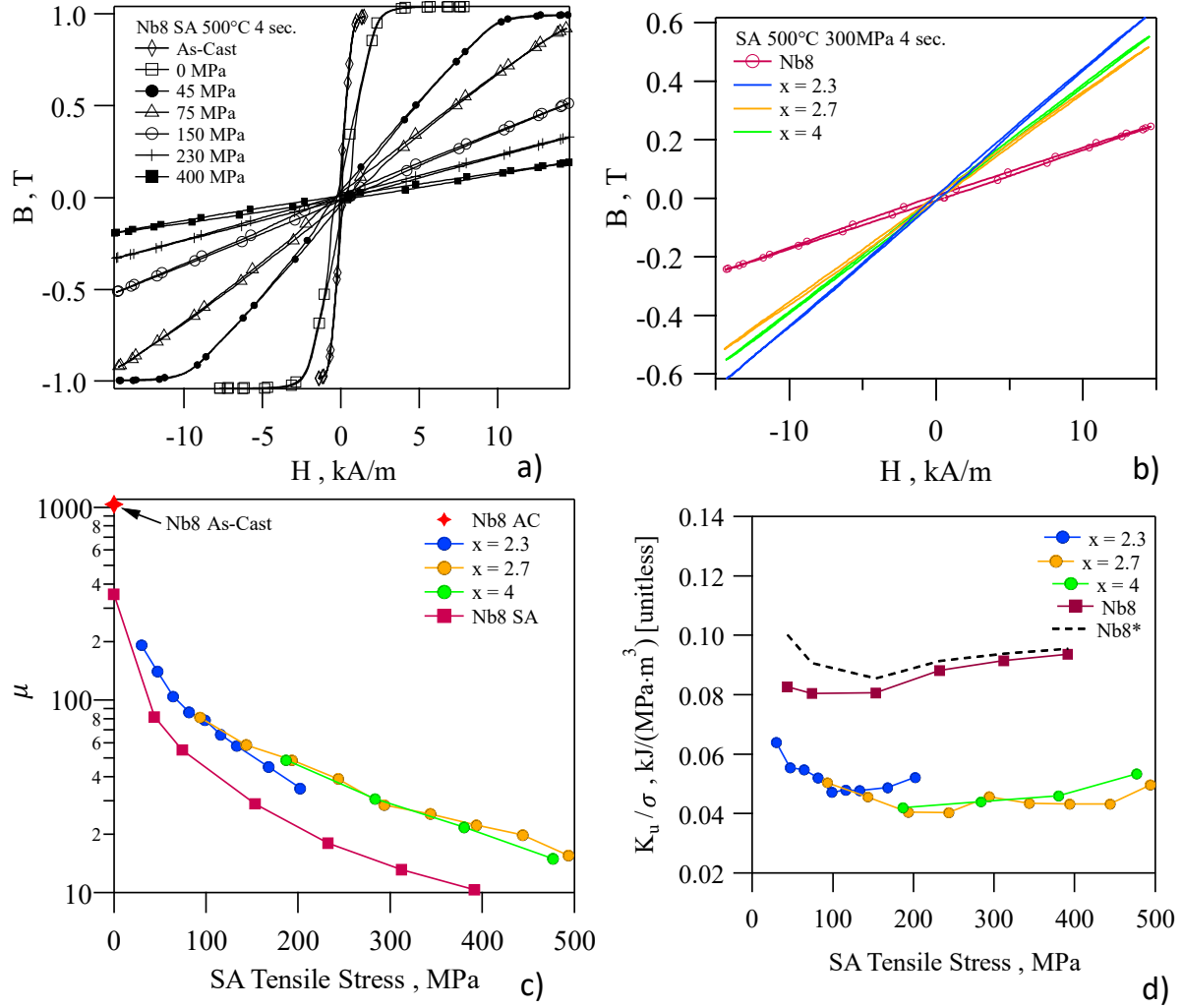


Figure 1: (a) BH loops of as-cast and annealed Nb8 alloy ribbon strips stress-annealed at 500°C for 4 seconds, (b) BH loops of $x = 2.3$, 2.7, 4, and Nb8 ribbon strips stress-annealed at 500°C for 4 seconds at 300 MPa, (c) Relative permeability, μ , vs. annealing stress, σ , in as-cast and stress-annealed strips at 500°C for 4 seconds, and (d) stress-induced K_u/σ vs. σ for $x = 2.3$, 2.7, 4, and Nb8 ribbon strips from (c). The Nb* curve represents the K_u/σ for the Nb8 ribbon using $K_{u,0}$ from as-cast ribbon, instead of that from the 0 MPa annealed condition. K_u data in (d) for $x = 2.3$, 2.7, and 4 ribbons are not corrected with respect to a 0 MPa annealed baseline. BH loops of strips were measured at 400 Hz.

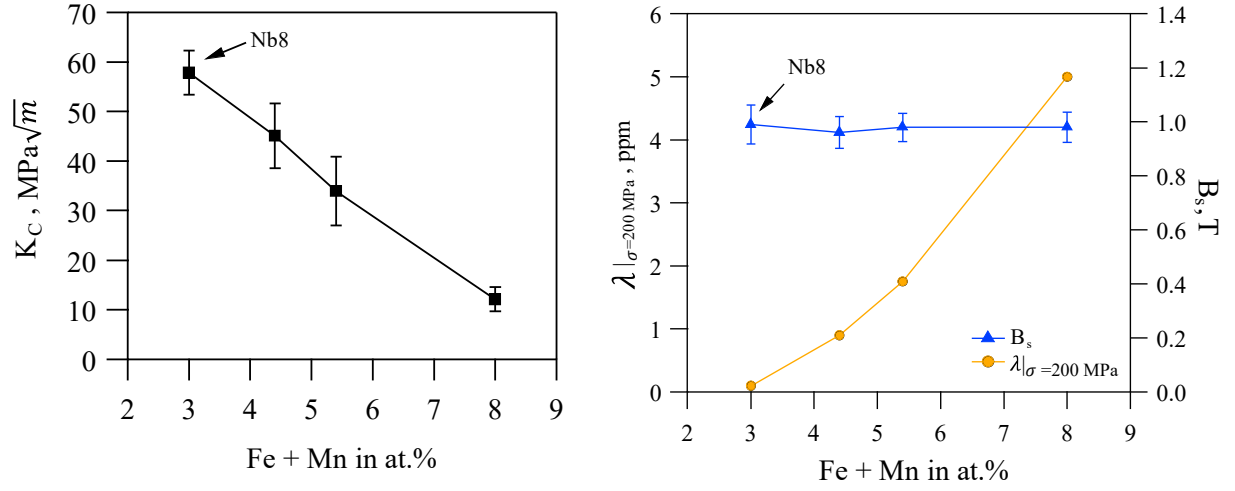


Figure 2: (a) Average fracture toughness, K_C vs. Fe + Mn concentration where error bars represent the standard error of the mean with 95% confidence and (b) bulk saturation magnetostriction, λ_s^{bulk} and saturation induction, B_s vs. Fe + Mn concentration for x = 2.3, 2.7, 4, and Nb8 ribbon strips stress-annealed at 500°C for 4 seconds under 200 MPa. Ribbon was excited at 400 Hz in (b).

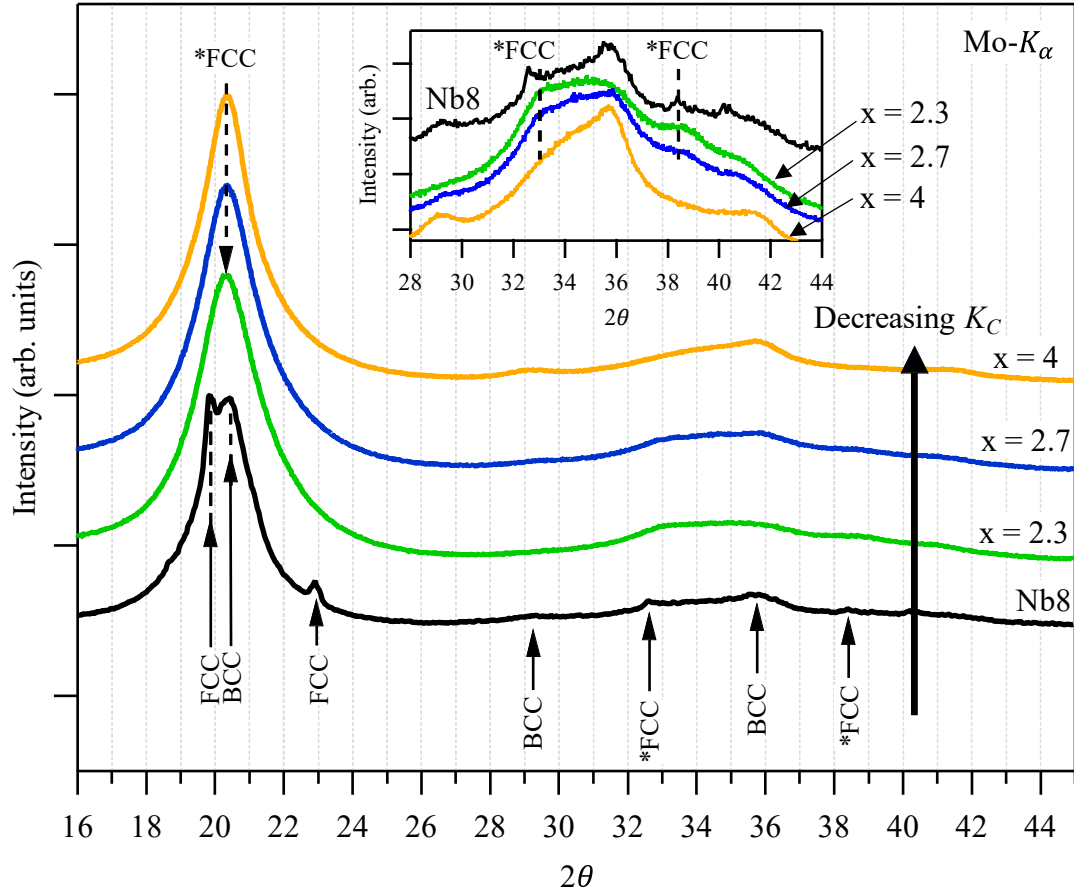


Figure 3: Room temperature X-ray spectra for $x = 2.3, 2.7, 4$, and Nb8 ribbon strips stress-annealed at 500°C for 4 seconds under 100 MPa. Spectra were vertically offset in decreasing order of fracture toughness, K_C . The inset depicts the spectra between a Bragg angle, 2θ , of 28 and 44 for clarity. The “*FCC” labels indicate HCP/FCC peaks corresponding to close-packed structures that are not affected by stacking faults. Unlabeled peaks from Nb8 ribbon correspond to the cubic 23:6 phase and light surface oxide [36].

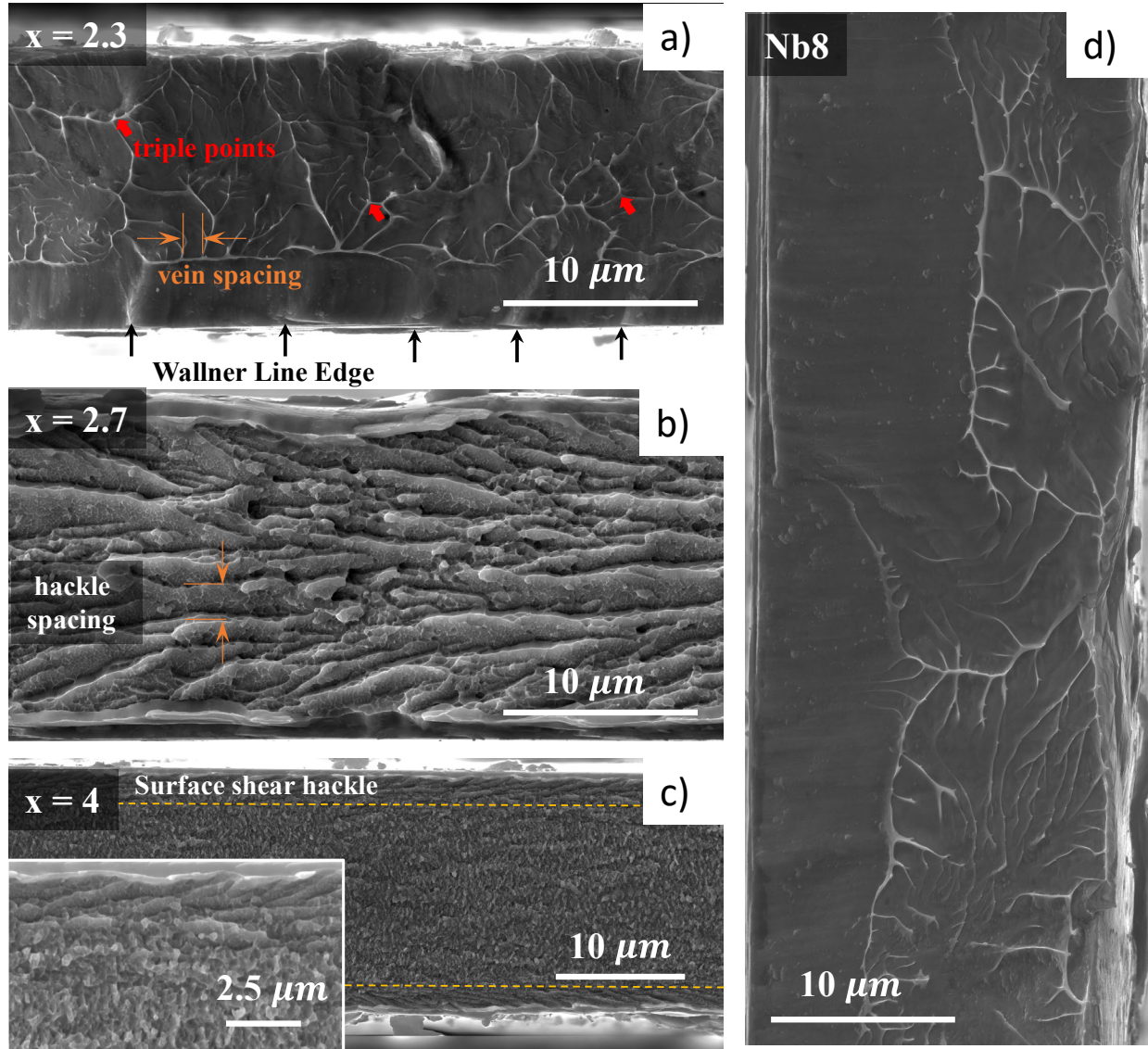


Figure 4: Fracture surfaces for (a) $x = 2.3$, (b) $x = 2.7$, (c) $x = 4$, and (d) Nb8 ribbon strips stress-annealed at 500°C for 4 seconds under 200 MPa. See text for description of the fracture morphologies.

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