Eutectic Solidification Limits and Mechanical Properties of Sm-Co-Fe Alloys

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EUTECTIC SOLIDIFICATION LIMITS AND MECHANICAL PROPERTIES
OF Sm-Co-Fe ALLOYS

by

Wendy Ann Wagner

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EUTECTIC SOLIDIFICATION LIMITS AND MECHANICAL PROPERTIES
OF Sm-Co-Fe ALLOYS

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Creating a permanent magnet with a higher energy product than existing materials is attractive in order to optimize magnetic performance. The eutectic microstructure of Sm-Co alloys is attractive for magnets since primary rods of the Co-phase can act as a soft magnetic phase in the matrix of the Sm$_2$Co$_{17}$ hard magnetic phase, forming two-phase magnets. Fe replacement in Sm-Co alloys provides an opportunity to maintain the desirable eutectic microstructure of Co$_{1-x}$Fe$_x$ rods embedded in a Sm$_2$(Co$_{1-x}$Fe$_x$)$_{17}$ matrix while improving the magnetization and lowering the cost. The purpose of this study is to determine the eutectic solidification limit of Sm$_8$(Co$_{1-x}$Fe$_x$)$_{92}$ alloys and their corresponding mechanical properties.

Samples were made with x from 0 to 1, in increments of 0.05 by arc melting followed by melt spinning at 10 m/s. Microstructural analysis revealed that the eutectic structure can be maintained up to x = 0.30 before the development Co/Fe dendrites. Compositional analysis found that Fe partitions to the Co/Fe rod phase. Magnetic analysis confirmed the increase in magnetization with increasing Fe content.

Mechanical testing revealed the hardness and relative strain at fracture of the alloys. The hardness increases to a maximum at x = 0.30 coinciding with the eutectic limit, and decreasing for x ≥ 0.35 due to the presence of dendrites in the microstructure.
Relative strain at fracture was determined from bend testing to reveal the increasing brittleness of the samples for \( x \leq 0.35 \). For \( x \geq 0.40 \), the relative strain at fracture was found to increase, then decrease, due to dendrites and then \( \text{Sm}_2\text{Co}_7 \) phase along grain boundaries, respectively.
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1. Introduction

1.1 Magnetic Materials

Magnets are a crucial component for many applications. They are used extensively in power generation components such as turbines and compressors, as well as in data storage applications. The demand for high performance magnets has increased substantially as the cost of materials increases and the need to reduce weight and/or size continues to be a driving factor in industry. To improve the properties of a magnet, the microstructure can be analyzed in order to understand how alloying and processing history affect the properties.

1.2 Objective

The eutectic microstructure of Sm$_8$Co$_{92}$ results in a desirable microstructure for magnetic applications [1]. One of the major limitations for this alloy in creating a higher energy product magnet is the anisotropy of the material which limits the coercivity. If a soft phase can be incorporated into the microstructure on a sufficiently fine scale, the remanence for the magnetic material can be increased while maintaining the high coercivity of the initial alloy, resulting in a higher energy product magnet. The mechanical properties are also of interest since varying applications require different hardness and strengths for magnetic materials. Samarium-cobalt based magnets are very
hard and brittle, so determining how the mechanical properties are affected is imperative to manufacturing and machining processes [2]. The objective of this research is to determine the eutectic solidification limit of Sm-Co-Fe alloys and their corresponding mechanical properties.

1.3 Phase Diagrams

Phase diagrams are crucial to understanding the microstructure of a material. Each phase in a material has different properties and by selecting the correct composition, different phases can be produced. The microstructure of interest for this study is the eutectic microstructure. A eutectic occurs when, upon cooling, a liquid transforms directly into a two-phase solid. Figure 1 shows a generic binary phase diagram containing a eutectic. At composition 1, upon cooling the material passes through A+L, where primary A dendrites form. The corresponding liquid phase becomes enriched in B until it reaches the eutectic point, at which a eutectic structure forms. The final microstructure will be primary-A dendrites surrounded by A+B eutectic. This type of microstructure is hypoeutectic. The composition at 2 passes directly through the eutectic so that only A+B eutectic is present in the final microstructure. At composition 3, phase B will form as dendrites and then A+B eutectic will surround the B phase. This type of microstructure is hypereutectic. Eutectic structures can be either lamellar or fibrous depending on the relative volume fractions of the two phases.
A eutectic forms in the Co-Sm phase diagram at 8 atomic % Sm (Figure 2). Upon solidification, the eutectic structure consists of FCC $\alpha$-Co rods in a Sm$_2$Co$_{17}$ matrix. The Sm$_2$Co$_{17}$ phase forms at $\sim$10.5 atomic % Sm. Both the rod and matrix phase form simultaneously. This is the microstructure that is desired for good magnetic properties.
Figure 2. A portion of the Co-Sm phase diagram. Eutectic at 8 atomic % Sm, with Sm$_2$Co$_{17}$ phase at ~10.5 atomic % Sm (after [3]).

If less Sm is present in the alloy, a hypoeutectic structure will form, where FCC $\alpha$-Co dendrites will be the first phase to form upon solidification, with the remaining liquid solidifying as the eutectic structure. If more Sm is in the alloy, a hypereutectic structure forms where the first phase to form upon solidification is the Sm$_2$Co$_{17}$ phase, with the remaining liquid solidifying as the eutectic structure.

While a eutectic does not exist in the Fe-Sm phase diagram, the Sm$_2$Fe$_{17}$ phase exists at ~10.5 atomic % Sm (Figure 3). A two-phase region is formed for 8 atomic % Sm consisting of BCC $\alpha$Fe and Sm$_2$Fe$_{17}$. 
Figure 3. A portion of the Fe-Sm phase diagram. Sm$_2$Fe$_{17}$ line compound appears at $\sim$10.5 atomic % Sm (after [3]).

The Co-Fe phase diagram indicates complete solubility at high temperatures for FCC $\alpha$-Co and FCC $\gamma$-Fe (Figure 4). From approximately 10 to 23 atomic % Fe at 500 $^\circ$C, a two-phase region consisting of FCC (Fe,Co) and BCC (Fe,Co) exists. From approximately 23 to 100 atomic % Fe at 500 $^\circ$C, solely BCC (Fe,Co) is present.
Since both the Sm$_2$Co$_{17}$ and Sm$_2$Fe$_{17}$ exist in the binary phase diagrams, one would expect that a complete solid solution of the formula Sm$_2$(Co$_{1-x}$Fe$_x$)$_{17}$ exists between the two [4]. How far the eutectic extends towards the Sm-Fe phase diagram is the objective of this study which a section of the ternary phase diagram demonstrates [5] (Figure 5).

Figure 4. Fe-Co phase diagram (after [3]).
1.4 Solidification

Controlling the solidification of an alloy will greatly affect its final properties. Solidification methods such as directional solidification, solid casting and rapid solidification will result in very different microstructures for the same alloy. Heat flow and temperature gradient are key components that can be manipulated once an alloy composition is chosen in order to influence the final microstructure.

During solidification with equilibrium cooling, the atoms have sufficient time to diffuse so that the most energetically favorable microstructure is formed. Non-equilibrium cooling, or normal solidification processes for that matter, produce various non-equilibrium micro-constituents. Rapid solidification utilizes high cooling rates, or large undercooling $\Delta T$ to increase the interface velocity. A large interface velocity, $v$, will reduce the scale of the microstructure since $v$ is proportional to $\Delta T^2$ [6].

For a eutectic alloy, the theoretical relationship between microstructural scale, $\lambda$, and growth velocity is [7]
\[ \lambda = \frac{2B\gamma T_e}{\Delta H_f R D_L} \]  

where \( B \) = constant, \( \gamma \) = surface energy, \( T_e \) = eutectic temperature, \( D_L \) = diffusivity of liquid, \( \Delta H_f \) = heat of fusion, and \( R \) = growth rate. For a given system, heat of fusion, \( B \), and \( T_e \), are constants. The surface energy will change as the scale of the microstructure changes, setting a lower limit for \( \lambda \), but this will not be discussed here. The liquid diffusivity is dependent upon temperature according to [6]

\[ D = D_o e^{-Q/(RT)} \]  

where \( D_o \) = material constant of diffusivity, \( Q \) = activation enthalpy (constant for a given material), \( R \) = universal gas constant, \( T \) = absolute temperature. So at lower temperatures, or larger undercooling, the diffusivity is reduced. The growth rate, \( R \), also changes with the undercooling according to [6]

\[ R = k_4 \Delta T^2 \]  

where \( k_4 \) is a constant and \( \Delta T \) is the undercooling. With larger undercooling, the growth rate increases. By providing a large undercooling, the growth rate increases and the diffusivity decreases. Since \( \lambda \propto \sqrt{1/R} \), a fine scale can be achieved [8].

It has been shown that higher wheel speeds retain the eutectic structure in the Sm-Co system, although the scale of the structure was reduced, with rod diameters reaching \( \sim 25 \) nm at 40 m/s (Figure 6) [9]. Figure 6 shows the relationship between (1) and wheel speed, which follows \( V^{-1/5} \) where \( V \) is the wheel speed. Thus, increasing wheel speed proportionately increases the growth rate.
1.5 Permanent Magnets

While extensive magnetic testing is not an integral part of this project, understanding the magnetic theory which drives this research is crucial. Permanent magnets continue to be of high interest as the need for them is only continuing to grow. Nd-Fe-B based magnets are currently the strongest magnet on the market with commercially available energy products as high as 48-55 MGOe, while Sm-Co based magnets have a maximum available energy product of 31.5 MGOe [10, 11]. Sm-Co-based magnets have the advantage of excellent high-temperature performance (up to 350 °C), whereas Nd-Fe-B magnets are limited to near-ambient temperatures.

The strength of a permanent magnet is characterized by \((BH)_{\text{max}}\), conventionally referred to as the energy product. To understand this measurement, a hysteresis loop for a magnetic material is show in Figure 7. A magnetic field or magnetizing force, \(H\), is
applied to a material and plotted on the abscissa. The magnetic flux density response of the material is recorded on the ordinate axis. The magnetic field is applied until the material reaches magnetic saturation. After the material has been brought to the condition of magnetic saturation, the magnetic field is reduced to zero, and the remaining flux density of the material is measured as the remanence. Reversing the magnetic field until the material retains no magnetic flux density, the coercivity of the material is found. The product of the remanence and the coercivity results in the energy product, \((BH)_{\text{max}}\) of the material.

Figure 7. Hysteresis loop showing the saturation magnetization, remanence, and coercivity (after [12]).
The remanence is a result of the intrinsic atomic properties of the elements in the alloy. The coercivity is derived from the anisotropy of the material. For soft magnetic materials, a low coercivity and high remanence are present resulting in a low energy product. For permanent magnets, a higher coercivity is present, but a reduced remanence is present. To improve the properties of permanent magnets, both a high coercivity and high remanence are desired.

1.6 Two-Phase Magnets

Since nano-composite or two-phase magnets were first suggested by Kneller and Hawig in 1991 [13], much research has gone towards achieving this type of magnet [14, 15, 16, 17]. The most common system for nanocomposite permanent magnets consists of a rare earth (RE) element such as neodymium or samarium, alloyed with transition metals (TM) iron or cobalt forming a granular structure. The soft phase in such a magnet must have low anisotropy (i.e. iron) in order to allow the hard phase, which has an highly anisotropic structure, to easily influence the soft phase. Experimentally, the volume fraction of the soft phase is found to be limited to approximately 15% in bulk materials [17].

Fibrous eutectic structures are a good candidate for producing an exchange-coupled magnet since the rods can be considered to act as the soft phase surrounded by the matrix hard phase. At the eutectic composition in the Sm-Co phase diagram, the resulting microstructure consists of FCC-Co rods embedded in a Sm$_2$Co$_{17}$ matrix. This microstructure is attractive since Sm$_2$Co$_{17}$ is a hard magnet and the Co-rods can act as a
softer phase. By replacing Co with Fe, the saturation magnetization will be increased. The scale of the eutectic microstructure must be appropriately fine as defined by the exchange length to result in exchange coupling between the hard and soft grains [18].

Permanent magnets can influence soft magnetic material within a certain physical distance defined as the exchange length,

\[ x = \pi \sqrt{\frac{A}{K}} \]  \hspace{1cm} (4)

where \( A \) is the exchange strength [erg/cm or J/m], and \( K \) is the anisotropy constant [ergs/cm\(^3\) or J/m\(^3\)] for a given material [1]. If a soft phase can be created within a hard phase so that the scale of the soft phase is not greater than twice the exchange length, the hard phase can completely influence the soft phase (Figure 8). Theoretically, the exchange length varies from 5 to 10 nm [13, 17]. Experimentally, however, soft grain sizes of \( \sim 30 \) nm have been found to produce effective exchange coupling [19]. In the material in this study, this means if Co-rods have diameters less than 30 nm, effective exchange coupling can be produced. The soft phase will increase the remanence of the material, while the hard phase will increase the coercivity so that a much higher energy product can be obtained by this two-phase magnet.
Figure 8. Hard and soft magnetic phases, demonstrating the influence of the hard phase on the soft phase on the scale of twice the exchange length.

The eutectic structure allows the scale of the Co rods to become sufficiently small so that a two-phase magnet with a higher energy product can be created with the rods acting as the soft phase. An added benefit of replacing Co with Fe is that the cost of Sm-Co magnets can be reduced.

1.7 Sm-Co Magnets

Sm-Co magnets are desirable for many applications because of their higher Curie temperature. At room temperature, the $\text{Sm}_2\text{Co}_{17}$ structure has the space group symmetry $\overline{R}3m$ with the prototypical $\text{Th}_2\text{Zn}_{17}$ structure. Pairs of cobalt atoms occupy specific lattice sites and are commonly referred to as Co dumbbells. In this structure, the so-called Co dumbbells occupy specific lattice sites (Figure 9), which results in a long-range order in the crystal structure. The long-range order can be suppressed by ternary alloying or by rapid solidification [20], resulting in formation of the metastable $\text{SmCo}_7$ structure which lacks the specified placement of Co dumbbells on known lattice sites resulting in the lack of the ordered x-ray diffraction peaks of the $\text{Sm}_2\text{Co}_{17}$ phase. The
focus of this project was not on the presence or absence of long range ordering of the lattice structure, so this phase will always be referred to as the Sm$_2$Co$_{17}$ phase in this paper.

Figure 9.  Crystal structure of the Sm$_2$Co$_{17}$ phase (after [21]).
2. Experimental Procedures

2.1 Sample Selection

Samarium-cobalt-iron alloys with nominal composition of Sm$_8$(Co$_{1-x}$Fe$_x$)$_{92}$ with x = 0.00 - 1.00 in increments of 0.05 were selected for this study.

2.2 Measuring Samples

The samples were massed on a digital balance to +/- 0.00001 g for each element according to their weight percent. Sample mass varied from 5 to 8 g to ensure enough ribbons were obtained for analysis. An extra 3 weight % Sm was added to compensate for vaporization loss during arc melting and melt spinning. The amount of extra Sm to add to account for Sm loss during melting was done by a trial-and-error method where different excess amounts of Sm were added. The sample masses at each stage were carefully monitored, and all sample weight loss was assumed to be due to Sm vaporization. The composition of the alloy after sample loss was back-calculated to verify correct sample composition. The calculated sample composition corresponded extremely well with observed microstructures. We thus determined the Sm loss for our specific melt practices, and alloy compositions adjusted accordingly.
2.3 Arc Melting

An arc is created by striking a current through a charged electrode to the metal. This process takes place in an ultra high purity (UHP) argon atmosphere. Each sample was loaded into the arc melting chamber, into a finger-shaped depression in a copper base, with a zirconium ingot placed in a separate depression to act as an oxygen getter. The chamber partially consists of a cylindrical section of glass to allow visual observation during melting. The chamber was evacuated to approximately 60 millitorr and flushed with UHP argon six times, to a final pressure of – 15 in Hg. The zirconium ingot was melted first to ensure the arc melter was operational and an inert atmosphere was present. If the zirconium ingot remains shiny after melting, the atmosphere is inert. The zirconium ingot was melted a second time to ensure residual oxygen was captured. The sample was then melted until a rough ingot was formed. The sample was turned over and melted a second time. The sample was turned over and melted a third and final time, forming a uniform shaped ingot.

2.4 Melt Spinning

Melt spinning is a rapid solidification process where molten metal is forced onto a spinning copper wheel, causing the metal to rapidly solidify and form thin metal ribbons. This additional step ensured homogeneity in the material, and the low wheel speed resulted in a microstructural scale readily observed by optical microscopy. The first step in the process was preparing the crucible with an orifice of 0.81 mm, and then massing the crucible. The crucible was massed before melt spinning in order to calculate actual
Sm loss after melt spinning. The sample was loaded into the crucible, and the crucible mounted in the melt spinner chamber. The chamber was evacuated and backfilled with UHP argon to a final pressure of 940 mbar. An ejection pressure of 200 mbar above chamber pressure was set to assist in ejecting the molten metal from the crucible. The speed of the copper wheel was set to 10 m/s. The sample was heated by radio frequency induction produced in the copper coil. Once the sample was molten, the overpressure was applied and the sample was forced through the crucible orifice onto the spinning copper wheel resulting in thin ribbons. After the melt spinning was complete, all ribbons and the post-melt spin crucible were retrieved and massed in order to calculated actual Sm loss, and thereby back calculating the nominal composition of the sample.

2.5 Powder X-Ray Diffraction

Powder X-ray diffraction is a technique used to identify phases present, unit cell parameters, crystal orientation, and atomic structure of the powdered material. Since X-rays have a wavelength on the order of $10^{-10}$ m they are useful when analyzing atomic-scale structures. X-rays will enter a material and interact with the electrons present on atoms. In most cases, this interaction produces destructive interference. However, when the X-rays interact and are in phase, constructive interference, or diffraction, occurs according to Bragg’s Law of Diffraction

$$n\lambda = 2d \sin \theta$$

where $n$ is the number of wavelengths, $\lambda$ is the wave length of the X-ray, $d$ is the interplanar spacing of the crystal, and $\theta$ is the incident angle.
Ribbons were gently powdered using a mortar and pestle and analyzed using a Rigaku Multiflex x-ray diffractometer with a Cu-Kα X-ray source. By powdering the sample, a polycrystalline material is produced so that diffraction occurs at all orientations of 2θ. The powdered sample was placed on a zero background slide and the diffracted X-rays detected with a moving detector. The diffraction patterns produced by each crystallographic phase are unique and were identified by comparing to JCPDS-PDF (Powder Diffraction File) cards, which are existing powder diffraction X-ray standards published by the International Centre for Diffraction Data.

2.6 Optical Microscopy

In binary Sm-Co, the presence of the eutectic is well-known, and so samples were observed with an optical microscope to search for off-eutectic microstructures, signifying that Sm loss altered the composition to be either hypo- or hypereutectic. Optical microscopy was also used to determine the eutectic solidification limit of the Sm-Co-Fe alloys. Optical microscopy was a key step in determining the appropriate amount of extra Sm to add to samples to account for Sm vaporization.

2.7 Electron Microscopy

Scanning electron microscopy is a technique used to observe the microstructure of a specimen with a resolution of approximately 1 nm. A beam of electrons is produced by the electron source and the electron beam is focused by passing through a series of magnetic condenser lenses. The electron beam is rastered across the sample at a chosen a
location, and the electrons interact with the sample causing secondary and backscattered electrons to be released, as well as X-rays. When the primary electron beam interacts with the sample there is a tear-shaped volume known as the interaction volume beneath the probe location from where the electrons and X-rays are emitted (Figure 10). The size of the interaction volume depends on the energy of the electron beam and the material being examined.

Secondary electrons are produced when a source electron passes near an atom in the specimen and imparts some of its energy to an outer-shell electron in the atom, so the atom’s electron is ‘knocked off’ the sample. These secondary electrons are produced by inelastic scattering, have low energy (2-5 eV) and provide topographical information.
They are detected and then sent to an amplifier which produces an image of the specimen. The brightness of the image depends on how many secondary electrons were detected.

Backscattered electrons are produced when the source electrons penetrate the sample, elastically collide with existing atoms and change direction, and ultimately leave the sample. Because the probability of scattering depends on the number of electrons in the sample, backscattered electrons provide atomic number information. The higher the atomic number of an element, the more backscattered electrons the element produces and are detected. So a higher atomic number results in a brighter image.

All samples were cold mounted using a thermosetting resin (epoxy) by placing the ribbons on the base of a cylindrical mold, then carefully applying the epoxy over the top. The epoxy was allowed to set for 24 hours, and then the mold removed leaving a cylindrical sample with the ribbons mounted on one end. The samples were polished on metallographic polishing wheels and then on an automatic polisher (MINIMET 1000) with alumina solutions down to 0.05 μm particle size. Samples were lightly etched with a 2% Nital solution to expose the microstructure.

To ensure conductivity for the electrons, the samples were then coated with a thin layer (~ 20 nm) of chromium by the method of sputter coating. Placing the samples in the Hitachi S4700 Field-Emission Scanning Electron Microscope (FE-SEM), the chamber was evacuated of air and the electron gun activated to emit high energy electrons at 10 kV. Micrographs of the samples were obtained from the secondary electrons detected by the FE-SEM.
2.8 X-Ray Microanalysis

X-rays are also emitted from the sample as a result of beam electrons producing inner shell ionization events. The ionization creates an electron vacancy that can be filled by an outer shell electron. As an outer shell electron falls into the vacancy, an X-ray is emitted and can be detected. X-rays can be measured either by energy dispersive spectroscopy (EDS) or by wavelength dispersive spectroscopy (WDS).

X-rays emitted have a characteristic energy specific to the element from which it originated, and this energy is detected and measured during energy dispersive spectroscopy. X-rays also have a characteristic wavelength specific to the element from which it originated. During wavelength dispersive spectroscopy, the emitted X-rays from the interaction volume are counted and the specific wavelengths from each element are detected and compared to known standards.

To determine composition of the different phases present in the samples, a JEOL JXA-8200 WD/ED Combined Microanalyzer Superprobe was used. Wavelength dispersive X-ray spectroscopy was used (Figure 11). Backscattered electron images were used to select points for analysis in the microprobe. Pure element standards were used for Co and Fe to determine alloy compositions. Since a pure Sm standard was unavailable, a SmAl standard was used. The SmAl had a 1:1 atomic ratio, and was annealed at 900 °C for 1 week to ensure homogeneity. Back-scattered electron images were used to select points for analysis in the microprobe, and the acceleration potential was 12 kV with probe currents of 25 - 30 nA.
Figure 11. X-rays emitted from interaction volume. Scale of microstructure may be finer than interaction volume.

2.8 Magnetic Analysis

Magnetic analysis was performed using a Quantum Design Magnetic Property Measurement System (MPMS) Superconducting Quantum Interference Device (SQUID) magnetometer at room temperature from 0 to 5 T.

2.9 Mechanical Testing

Mechanical testing was conducted on the samples to determine select physical quantities: Knoop Hardness and Relative Strain at Fracture which is related to ductility.
2.9.1 Knoop Hardness Testing

Knoop Hardness testing is used on very small or selected areas of a specimen. A diamond shaped indenter is forced onto the sample for a specified period of time, and the resulting indentation size is measured in filar units through a microscope. This is converted to a Knoop Hardness (HK) value according to

\[ HK = \frac{14229 \times L}{(FU \times c)^2} \]  

where \( L \) = load in grams, \( FU \) = measured indentation in filar units, and \( c \) = calibration from the microscope objective.

To prepare the samples for hardness testing, the ribbons were held by a specimen clip so that the cross section was exposed (Figure 12). The samples were then cold mounted in epoxy and polished on metallographic polishing wheels and then on an automatic polisher (MINIMET 1000) with alumina solutions down to 0.05 μm particle size.

![Specimen Clip and Ribbon](image)

Figure 12. Ribbon cross section exposed after cold mounting.

Samples were tested on a Wilson Tukon Knoop Hardness tester with a 50 g load and a dwell time of 2 seconds. Objective C was used to measure indentations resulting in
a calibration of 0.2469 μm. Approximately 20 tests per sample were completed, resulting in a standard deviation of ~450 HK.

2.9.2 Bend Testing

Bend testing is a method used to characterize embrittlement of nanocrystalline ribbons [23, 24]. The ribbon is placed on a base between two plates which are slowly pressed together so that the ribbon begins to make an upside down ‘U’ shape (Figure 13 and Figure 14).

Figure 13. Bend test apparatus. Plate 1 and base are connected and stop the ribbon at their junction. The base slides underneath stationary plate 2 while keeping the ribbon between both plates.
The distance between the plates at fracture is measured and used to calculate the relative strain at fracture which is defined as

\[
e_{f_{\text{Brittle}}} = \frac{d}{\frac{D^2 + 3L^2}{\sqrt{4(3L^2 - 3D^2) - d}}}
\]

(7)

\[
e_{f_{\text{Ductile}}} = \frac{d}{D-d}
\]

(8)

where \(d\) is the ribbon thickness, \(D\) is the distance between the plates at fracture, and \(L\) is the original ribbon length [25]. Ribbons were defined as ductile or brittle based on the geometry at fracture according to

\[
D > \frac{2L}{\pi}; \quad \text{Brittle Sample}
\]

\[
D \leq \frac{2L}{\pi}; \quad \text{Ductile Sample}
\]

(9)
Each sample had thickness measured with a digital micrometer, and length measured with digital calipers. Samples were placed with the wheel side of the ribbon normally down towards the base. A Thor Labs translation stage was used to push the plates together at a velocity of 0.250 mm/s while continually monitoring displacement. A digital video camera was used to record each test to determine distance between the plates at fracture.

Errors in bend testing are possible due to the resolution of the digital video and displacement tracking when capturing the exact fracture location of the ribbons. Also, the initial ribbon length for some alloy compositions was substantially less than most ribbons, making measurements difficult.
3. Results and Discussion

3.1 Basic Microstructure

At the nominal composition of Sm$_8$Co$_{92}$ a eutectic structure forms, with Sm$_2$Co$_{17}$ forming the matrix phase and primary-Co rods as the secondary phase (Figure 15). Melt spinning at 10 m/s produced a fairly coarse microstructure with an average rod diameter of 300 nm.

![Figure 15. Scanning electron micrograph of Sm$_8$Co$_{92}$ showing the eutectic structure consisting of primary Co rods in a Sm$_2$Co$_{17}$ matrix.](image)

To determine the extent of eutectic structure formation in Sm$_8$(Co$_{1-x}$Fe$_x$)$_{92}$ alloys, melt spinning was done at 10 m/s to allow microstructural observation by optical microscopy. An entirely eutectic structure was observed at x=0.3 (Figure 16(a)). At x=0.35, primary dendrites were observed (Figure 16(b)). The dendrites become more predominant at higher Fe content, and at x=0.6 the eutectic structure is entirely lost (Figure 16(c)).
3.2 X-Ray Diffraction

For the ribbons melt spun at lower wheel speeds (10 m/s), directional growth of the eutectic colonies was observed (Figure 17). X-ray diffraction patterns of the wheel and non-wheel sides revealed the presence of preferred growth (x-ray penetration is approximately 10 μm). The wheel side of the ribbon shows some texture in the [111] direction for the Co rods. The non-wheel side of the ribbon shows some texture in the <200> direction for the Co rods, and some texture in the [110] direction for the Sm<sub>2</sub>Co<sub>17</sub> phase (Figure 18).
Figure 17. Cross-sectional microstructure of Sm$_8$Co$_{92}$ melt spun at 10 m/s.

Figure 18. X-ray diffraction analysis comparing wheel side and non-wheel side of ribbons, showing texture in the [110] direction of the Sm$_2$Co$_{17}$ phase and texture in the <200> direction of the Co rod phase for the non-wheel side.
The x-ray diffraction results also revealed structural changes as the Fe content increased. At $x<0.15$, diffraction peaks corresponding to Sm$_2$Co$_{17}$ and FCC Co were observed (Figure 19 and Table 1). It is important to note that no peaks associated with long-range dumbbell ordering of the Sm$_2$Co$_{17}$ structure were observed (the most intense of which occurs at $2\theta \sim 38^\circ$). While the absence of the superlattice peaks suggests that the structure is closer to the TbCu$_7$-type, some weak order may exist and we did not specifically focus on its determination. Therefore, we will refer to the matrix phase as Sm$_2$Co$_{17}$ throughout this manuscript. The presence of FCC Co rather than HCP Co suggests that even modestly fast solidification rates can stabilize the FCC structure. With increasing Fe content, the presence of the FCC-Co decreases and the appearance of BCC-Fe was observed, first observed at $x=0.15$. This is consistent with the Co-Fe phase diagram, where above $x \sim 0.1$ a two-phase FCC+BCC region exists (Figure 4) [3]. At $x = 0.20$, the FCC-Co peak present at $2\theta = 51.50^\circ$ is barely evident and disappears completely by $x = 0.25$ (Figure 19). The disappearance of the FCC Co peak is expected by $x = 0.30$ since the phase boundary in the binary Fe-Co diagram occurs at $\sim 28$ atomic % Fe. As noted above, both the Sm$_2$Co$_{17}$ and Sm$_2$Fe$_{17}$ exist in the binary phase diagrams, so one would expect that a complete solid solution of the formula Sm$_2$(Co$_{1-x}$Fe$_x$)$_{17}$ exists between the two. Indeed, with increasing Fe content the Sm$_2$Co$_{17}$ peak shifts from $2\theta = 43.40^\circ$ at $x = 0.0$, to $2\theta = 42.82^\circ$ at $x = 1.0$, revealing only Sm$_2$Fe$_{17}$ and BCC-Fe peaks at $x = 1.0$. 
Figure 19. X-ray diffraction patterns showing the shift from the \( \text{Sm}_2\text{Co}_{17}/\text{Co} \) phases to the \( \text{Sm}_2\text{Fe}_{17}/\text{Fe} \) phases.

Table 1 FCC Co peaks identified as the (111) and (200) planes.

<table>
<thead>
<tr>
<th>Structure</th>
<th>Calculated Planes</th>
<th>2θ, deg. (Calculated)</th>
<th>2θ, deg. (Observed)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Co (HCP)</td>
<td>100</td>
<td>41.59</td>
<td></td>
</tr>
<tr>
<td>Co (FCC)</td>
<td>111</td>
<td>44.26</td>
<td>44.30</td>
</tr>
<tr>
<td>Co (HCP)</td>
<td>002</td>
<td>44.52</td>
<td></td>
</tr>
<tr>
<td>Co (HCP)</td>
<td>101</td>
<td>47.44</td>
<td></td>
</tr>
<tr>
<td>Co (FCC)</td>
<td>200</td>
<td>51.57</td>
<td>51.50</td>
</tr>
<tr>
<td>Co (HCP)</td>
<td>102</td>
<td>62.55</td>
<td></td>
</tr>
<tr>
<td>Co (HCP)</td>
<td>003</td>
<td>69.25</td>
<td></td>
</tr>
<tr>
<td>Co (FCC)</td>
<td>220</td>
<td>75.93</td>
<td></td>
</tr>
</tbody>
</table>
3.3 X-Ray Microanalysis

While Fe replaces Co in the microstructure, it is not known whether Fe preferentially partitions to the Co\(_{1-x}\)Fe\(_x\) or Sm\(_2(\text{Co}_{1-x}\text{Fe}_x)_{17}\) phases. Using a JEOL JXA-8200 WD/ED Combined Microanalyzer Superprobe, the composition of selected sample locations was determined. Line scans were also completed across select areas using a step size of 0.5 μm. Due to the small scale of the microstructure, some error was expected due to the interaction volume encompassing multiple phases. However, one-sigma uncertainty was routinely less than +/- 1% of the measured value. In the matrix phase, Sm content ranged from 9.91 atomic % to 9.92 atomic %, while the Fe+Co content ranged from 90.07 to 90.29 atomic % (Table 2). The stoichiometric composition is 10.53 and 89.47 atomic percent, respectively, but the phase diagram indicates a solubility range. Here, it appears that the composition of the matrix phase, with respect to Sm and total transition metal content, is constant over all values of x.

Table 2 Theoretical and measured alloy compositions.

<table>
<thead>
<tr>
<th>Sample x</th>
<th>Sm</th>
<th>Matrix Co</th>
<th>Co</th>
<th>Fe</th>
<th>Co</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.30</td>
<td>9.91</td>
<td>64.73</td>
<td>25.34</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.35</td>
<td>9.70</td>
<td>60.09</td>
<td>30.19</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.45</td>
<td>9.78</td>
<td>50.72</td>
<td>39.48</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.55</td>
<td>9.92</td>
<td>50.43</td>
<td>39.64</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Figure 20. Rod analysis demonstrating Fe preferentially alloying with the primary rod phase by an additional 0.05 for $x \leq 0.45$. For $x = 0.55$, the trend of Fe preferentially alloying changes due to increasing occurrence of dendrites. Expected composition determined from $\text{Co}_{1-x}\text{Fe}_x$. Fe (●, − − −), Co (♦, − − − − − − −).
Figure 21. Matrix analysis demonstrating corresponding Fe loss due to preferential alloying with rod phase for $x \leq 0.45$. For $x = 0.55$, trend changes due to increased occurrence of dendrites. Expected composition determined from $\text{Sm}_2(\text{Co}_{1-x}\text{Fe}_x)_{17}$. Fe (●, − − −), Co (♦, −−−−−), Sm (▲).

The Co:Fe ratio in both the matrix $\text{Sm}_2(\text{Co,Fe})_{17}$ and Co(Fe) rods differs from the nominal composition of the alloys, indicating that Fe is not evenly distributed between the two phases. For $x \leq 0.45$, Fe preferentially alloyed with the rod phase so that $x$ was consistently 5 atomic % higher than the nominal Fe content of the alloy (Table 2, Figure 20). Likewise, the Fe content in the 2-17 phase was lower than the nominal composition of the alloys. Correspondingly, the Fe content in the 2-17 phase was 0.79 to 1.5 atomic percent lower than the nominal composition of the alloys. Estimating the volume fractions of phases present from the Sm-Co binary phase diagram, the matrix phase was expected to have approximately 2% less Fe, which is on the order of what was observed experimentally (Figure 21).
Figure 20 and Figure 21 plot the “expected” Fe and Co content if Fe partitions equally to both phases (dashed lines), and the experimentally determined content of each phase. The deviation from the nominal Fe content was remarkably consistent up until \( x = 0.45 \). After \( x = 0.45 \), however, the Fe content in both the rod and matrix phases levels out, resulting in lower-than-expected Fe content in both phases, and consequently more Co in each as well. This change is due to the development of dendrites. The dendrites are Fe-rich and solidify first. Thus, the remaining liquid that ultimately forms the eutectic structure is relatively Fe-poor, resulting in lower Fe content for both phases in the eutectic. At \( x > 0.55 \), an additional phase was observed in the BSE image along grain boundaries and was revealed by probe analysis and line scan analysis to be the \( \text{Sm}_2(\text{Co}_{1-x}\text{Fe}_x)_7 \) phase. This further confirms a transition to peritectic solidification and the loss of the eutectic point in the ternary Sm-Co-Fe alloys.

Line scan analysis was also completed on select areas, confirming the trend for Fe to preferentially alloy with the rod/dendrite phase. Three large colonies of the Fe/Co dendrites can be seen in Figure 22. The matrix phase still consists of the eutectic structure, but the scale of the rods is much smaller than the Fe/Co dendrite colonies and is not resolved in this micrograph. The first 8 \( \mu \text{m} \) of the scan are across the eutectic region and confirm the composition to match \( \sim 8 \) atomic % Sm, with \( \sim 41 \) atomic % Fe and \( \sim 52 \) atomic % Co as expected due to the fine scale of the eutectic and the size of the interaction volume (Figure 23). As the line scan continues across one of the Fe/Co dendrite colonies, it can be seen that Fe preferentially alloys with the dendrite/rod phase by \( x_{\text{measured}} = x + 0.05 \).
Figure 22. Scanning electron micrograph of Sm$_8$(Co$_{0.55}$Fe$_{0.45}$)$_9$ displaying location of line scan across Fe/Co dendrite.

Figure 23. Line scan analysis demonstrating higher Fe concentration in dendrite phase than in matrix phase.
3.3 Magnetic Measurements

The magnetizations of the Sm$_8$(Co$_{1-x}$Fe$_x$)$_9$ alloys were determined at 5 T for alloys up to $x = 0.35$ (Figure 24). Because of its higher moment [6], Fe substitution is expected to increase the magnetization. The magnetization increases from 11.8 kGauss (1.18 T) to 14.0 kGauss (1.40 T) as Fe replacement increases to $x = 0.30$, an increase of 18.6%. The dramatic increase in magnetization for $x = 0.35$ is caused by the presence of dendrites in the alloy, which increases the volume fraction of the soft magnetic phase.

Figure 24. Magnetization at a field of 5 T revealing increasing magnetization with increasing Fe content.
3.4 Mechanical Properties

The mechanical properties of the ribbons were evaluated from the Knoop hardness (HK) and relative strain at fracture.

3.4.1 Knoop Hardness

Knoop hardness testing was completed on the samples with the results shown in Figure 25. There is a trend for the hardness to increase while the eutectic microstructure is present, with a decrease in hardness once dendrites form in the microstructure. While the standard deviation for each point plotted is ~450, the overall trend in the data is preserved. Binary Sm-Co alloy has hardness of 1867 HK.

At x = 0.05, there is a marked increase in the hardness to 2775 HK. This is thought to occur because of lattice strain caused by the Fe. That the hardness remains higher than the binary Sm$_8$Co$_{92}$ alloy for $0.10 \leq x \leq 0.30$ is thought to be because of the appearance of BCC Fe precipitates in the rods since the two phase region of BCC+FCC on the Co-Fe phase diagram occurs at x ~ 0.1. The hardness continues to increase with increasing Fe content to a maximum of 2968 HK at x = 0.30, an increase of 59%. The maximum hardness also coincides with the eutectic limit. For $x \geq 0.35$, the hardness decreases, mainly because of the occurrence of dendrites in the microstructure.
Figure 25. Knoop Hardness of alloys demonstrating an increase in hardness with increasing Fe content. Maximum hardness occurs at $x = 0.30$, where the eutectic is still intact.

### 3.4.2 Bend Testing

The relative strain at fracture was determined for the samples using bend testing. All samples were determined to be brittle from the condition given in Equation (9). For $x = 0.05$ and 0.10, the relative strain at fracture reveals a less brittle sample which is in agreement with the lower HK for these samples (Figure 26). For $0.15 \leq x \leq 0.30$, the relative strain at fracture decreases indicating increasing brittleness, again in agreement with the increasing HK. The increase for $0.40 \leq x \leq 0.45$ is due to the presence of dendrites which are large in comparison with the fine scale of the eutectic microstructure.
The decrease for $x \geq 0.50$ can be explained by the occurrence of the Sm$_2$Co$_7$ phase along grain boundaries, causing the samples to become much more brittle (Figure 27).

Figure 26. Relative strain at fracture for alloys.
Figure 27. Scanning electron micrograph of Sm$_8$(Co$_{0.45}$Fe$_{0.55}$)$_{92}$. Dark areas are Co-Fe dendrites, grey area is Sm$_2$(Co,Fe)$_{17}$ matrix phase, and bright/light areas are Sm$_2$Co$_7$. 
4. Conclusions

The determination of eutectic solidification limits of $\text{Sm}_8(\text{Co}_{1-x}\text{Fe}_x)_{92}$ alloys was completed. By defining the limit for Fe replacement in Sm-Co alloys, the composition and materials processing requirements and mechanical characteristics for creating a successful two-phase magnet are now better understood. The replacement of Co with Fe maintained the desired eutectic microstructure of a matrix of $\text{Sm}_2(\text{Co}_{1-x}\text{Fe}_x)_{17}$ and rods of $\text{Co}_{1-x}\text{Fe}_x$ for $x \leq 0.30$. From $0.35 \leq x \leq 0.55$ both Co/Fe rods and dendrites were present. For $x \geq 0.60$ only Co/Fe dendrites were present. Fe preferentially alloys with the rod phase by an additional 0.05 for $x \geq 0.45$. The addition of Fe results in an increase in magnetization from 11.8 kG to 14.0 kG for $0.00 \leq x \leq 0.30$, as expected. Mechanical testing revealed the hardness and relative strain at fracture for the various microstructures. Relative strain at fracture was $< 0.001 \text{ ef}$, indicating that all samples were brittle.
5. Bibliography


