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ALLOY DESIGN OF $\text{Nd}_2\text{Fe}_{14}\text{B}$
PERMANENT MAGNETS

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January 2000

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LABORATORY TECHNOLOGY RESEARCH PROGRAM

**CRADA FINAL REPORT-
ALLOY DESIGN OF $\text{Nd}_2\text{Fe}_{14}\text{B}$ PERMANENT MAGNETS**

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January 2000

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ALLOY DESIGN OF $\text{Nd}_2\text{Fe}_{14}\text{B}$ PERMANENT MAGNETS

J. A. Horton and J. W. Herchenroeder

ABSTRACT

This report summarizes the results of research conducted as part of CRADA ORNL94-0279 between Oak Ridge National Laboratory and Magnequench International, Inc.. The objective was to gain a better understanding of the fracture process in the rare earth based permanent magnets in order to possibly improve the mechanical properties of these inherently brittle intermetallics. The first part of the study established a technique for measuring a critical property, fracture toughness, and surveying currently available commercial material made by a number of different processes. The second part examined the effect process variables have on fracture toughness. In the third part, a number of composition variations were tried both as powder additives and as alloy modifications within the constraint of less than two volume percent additive so as not to significantly reduce the magnetic properties. While toughness improvements of $0.5 \text{ MPa}\sqrt{\text{m}}$ resulted from some additions, it was not enough to warrant production changes. At this point, a more fundamental approach was taken by examining cleavage fracture in single crystals, and more thoroughly examining the grain boundary structure and actual fracture modes. Related investigations, involving a microstructural analysis of an improved ribbon resulting from production changes and hot pressing of gas atomized powder were also explored.

OBJECTIVES

Neodymium magnets based on $\text{Nd}_2\text{Fe}_{14}\text{B}$ have the highest energy product of any permanent magnet. Since commercial production started in about 1988, these magnets have made rapid progress in their use in commercial applications such as sensors, stepper motors, computer hard disk drives and in permanent magnet motors. While most magnetic materials are rather brittle, it would be desirable if they had better mechanical properties. For example, better mechanical properties would lower production losses due to chipping and would, in some cases, allow a different magnetic design if the magnet component could also be a structural element. Few previous investigations have concentrated on mechanical properties of magnets. Because of the obvious energy savings associated with the use of

permanent magnets, DOE has shown an increased interest in permanent magnet research. This project has the additional benefit of utilizing techniques and expertise developed from a DOE program on structural intermetallics and applied it to a related and economically important group of materials.

BENEFITS TO DOE'S MISSION

Improving the fracture toughness of neodymium permanent magnets would increase use of these magnets in some of the more demanding applications resulting in energy savings from using permanent magnet motors and actuators instead of inductive devices. For example, one prototype electric vehicle drive motor made by a Magnequench partner spins a magnetic cylinder at 13000 rpm with an air gap of less than 1 mm. The failure rate due to strength and toughness limits, however, is currently too high for commercial production.

TECHNICAL DISCUSSION

The following sections are based on the contents of 5 published papers. The section entitled Process Variables and Alloy Modifications is based on unpublished work.

Fracture Toughness of Commercial Magnets- Summary [1-3]

$\text{Nd}_2\text{Fe}_{14}\text{B}$ is currently the best (highest energy product) hard magnet available. Like most permanent magnetic materials, it is rather brittle. The fracture toughness of commercially available hard magnet specimens were measured using a chevron notch, three-point bend test. Values for $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnets ranged from 2.5 to 5.5 $\text{MPa}\sqrt{\text{m}}$. By comparison, SmCo_5 was also measured and has a fracture toughness of 1.9 $\text{MPa}\sqrt{\text{m}}$. It appears that this testing technique will detect differences in fracture toughness between alloys with different compositions and fabricated by different production methods and should be able to track the fracture toughness as improvements are made to a particular alloy.

Since its development [4-5] in 1983, the production and use of $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnets has grown rapidly. While $\text{Nd}_2\text{Fe}_{14}\text{B}$ is not quite as brittle as the previous highest energy product magnet, SmCo_5 , it is still rather brittle. Due to the brittleness, accurate dimensions must be achieved by grinding rather than by conventional machining. In some demanding magnetic applications such as electric vehicle drive motors in which high rpm and small air gaps are necessary, the $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnets need to be used as a structural element and thereby oper-

Fig. 1. Geometry of chevron-notch bend specimen.

no slip deformation modes. In the optimized magnetic condition, $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnets are multiphase with some nonmagnetic grain boundary phases present. The amount, type, and morphology of these second phases affect both the magnetic and mechanical properties. Baseline fracture toughness measurements are needed to aid development of tougher materials. For this reason, techniques developed for other brittle intermetallic alloys were applied to this commercially important intermetallic alloy

Fracture Toughness of Commercial Magnets- Experimental Procedure

Eight different types of $\text{Nd}_2\text{Fe}_{14}\text{B}$ commercial magnets were used in this investigation. Fracture toughness was measured using a 3-point bend test. The chevron notches in the bend bars were wire electrodischarge machined (EDM), see Fig. 1. During the 3-point bend test, the crack starts in the apex of the triangular cross section of material. As the crack propagates, the length of the crack front increases, which decreases the chance for catastrophic failure and can often result in controlled crack propagation, as shown in Fig. 2, depending on the stiffness of the testing equipment. A span of 35 mm and a notch angle of 36 or 45° were used in the tests. The tests were performed in an Instron 4501 screw driven machine. The cross-head speed was 1 $\mu\text{m/s}$. Fracture surfaces were photographed in a field emission scanning electron microscope (SEM).

Fig. 2. Load displacement curve of a Sumitomo 42 specimen.

ate under substantial mechanical stress [6]. Any improvement in their fracture resistance will reduce the failure rates during use.

Commercially, the material is produced either by a cast, crush, and sinter process or by a melt spin and hot press process. Some recent references provide excellent reviews [7-8]. $\text{Nd}_2\text{Fe}_{14}\text{B}$ has a large unit cell, low symmetry, and

$\text{Nd}_2\text{Fe}_{14}\text{B}$ magnets produced by a cast, crush, and sinter process and manufactured by Sumitomo, Vacuumschmelze, and Shin-Etsu were obtained as blocks $5 \times 5 \times 40$ mm from Magnet Sales and Manufacturing, Inc., Culver City, CA. The advertised energy products ranged from 28 to 45 MGOe. For comparison purposes, specimens of SmCo_5 and Alnico were also obtained from Magnet Sales. Three types of melt spun material were obtained from Magnequench International. In the Magnequench process, rapid solidification in the form of melt spinning is used to produce fine grained materials. After a slight crushing, the powder is cold compacted and hot pressed to produce an isotropic magnet, known commercially as MQ2. A further die upsetting operation will take an MQ2 magnet and produce an anisotropic MQ3 magnet.

Fracture Toughness of Commercial Magnets- Results and Discussion

Controlled crack propagation occurred in all of the crushed and sintered specimens tested. A typical load displacement curve is shown in Fig. 2. The fracture toughness, K_{IC} , is essentially a measure of the absorbed energy during crack propagation, where the absorbed energy is the area under the load displacement curve in Fig. 2. K_{IC} was calculated [9] by an area method according to:

$$K_{IC} = \left[\frac{W_f}{A} \times E' \right]^{1/2}, \quad (1)$$

where W_f is the area under the load-displacement curve, A is the area swept by the crack, and E' is the plane strain Young's modulus given by:

$$E' = \frac{E}{(1 - \nu^2)}. \quad (2)$$

Poisson's ratio, ν , was assumed to be 0.25 and a value of 157 GPa was used for E for $\text{Nd}_2\text{Fe}_{14}\text{B}$, 150 GPa for SmCo_5 , and 193 GPa for Alnico. In the case of the MQ2 and one set of the MQ3 specimens, controlled crack propagation did not occur. In this case, K_{IC} was calculated by both a peak load method [10] and the area method. Similar results were obtained. The geometry and test method used here has been successfully applied to other intermetallics [11] and to ceramics [9-10].

Table I is a summary of the fracture toughness measurements. (An earlier version with rather preliminary data was published previously [1].) Clear and consistent differences were seen between the three manufacturers producing $\text{Nd}_2\text{Fe}_{14}\text{B}$ material by the cast, crush, and sinter method. These five sets of material exhibited controlled crack propagation. No orientation difference was observed between specimens fractured parallel or nor-

Table 1 Fracture Toughness, K_{IC} , of Commercial Permanent Magnet

	Process	Manufacturer	Grade	K_{IC} , MPa $m^{1/2}$	No. of Tests
SmCo ₅		Vacuumschmelze	18	1.9 ± 0.2	5
Nd ₂ Fe ₁₄ B	Spin Cast, Hot Press	Magnequench, MQ2	E1	2.5 ± 0.2	8
Nd ₂ Fe ₁₄ B	Spin Cast, Die Upset	Magnequench, MQ3	E1	2.7 ± 0.5	6
Nd ₂ Fe ₁₄ B	Sintered	Shin-Etsu	45	3.9 ± 0.2	7
Nd ₂ Fe ₁₄ B	Sintered	Sumitomo	28	3.9 ± 0.3	5
Nd ₂ Fe ₁₄ B	Sintered	Sumitomo	40	4.4 ± 0.2	6
Nd ₂ Fe ₁₄ B	Spin Cast, Die Upset	Magnequench, MQ3*	H	4.4 ± 0.3	8
Nd ₂ Fe ₁₄ B	Sintered	Vacuumschmelze	32	5.5 ± 0.8	5
Nd ₂ Fe ₁₄ B	Sintered	Vacuumschmelze	42	5.5 ± 0.2	8
Alnico		TDK		13.3 ± 0.9	5

*die upset by Daido Steel Co.

mal to the easy magnetization direction. Fracture toughnesses were calculated using the area method described above and yielded values ranging from 3.9 ± 0.2 MPa $\sqrt{m}$ for the Shin-Etsu 45 material to 5.5 ± 0.2 MPa $\sqrt{m}$ for the Vacuumschmelze 42 material.

The first two groups of MQ specimens in Table 1 from Magnequench fractured catastrophically. The MQ2 (hot pressed) specimens yielded an average value of 2.5 ± 0.2 MPa $\sqrt{m}$ while the MQ3 (hot pressed and die upset) specimens yielded values of 2.7 ± 0.5 MPa $\sqrt{m}$. The third entry for MQ material in the table used an H grade (Magnequench's commercial designation) of powder and was die upset by Daido Steel Co. The fracture toughness of this material was measured to be 4.4 ± 0.3 MPa $\sqrt{m}$, comparable to the cast and sintered material from Sumitomo.

For comparison purposes, specimens of SmCo₅ were tested and found to have a toughness of 1.9 ± 0.2 MPa $\sqrt{m}$. Alnico, using the same specimen geometry and method of calculation showed a fracture toughness of 13.3 ± 0.9 MPa $\sqrt{m}$, while the toughness of alumina ranges from 3 to 4 MPa $\sqrt{m}$.

Fracture surfaces were examined by SEM. Figure 3 shows fracture surfaces of MQ3 and Fig. 4 shows fracture surfaces of Vacuumschmelze 42, Sumitomo 40, and Shin-Etsu 45. The crushed and sintered material has much larger grains and larger amounts of various second phases. Multiple cracks formed during fracture in some of these phases in the Vacuumschmelze magnet suggesting that more energy was absorbed during the fracture process and this correlates with the higher toughness of this material. The much smaller grain size in the melt spun material is apparent in Fig. 3. The strip with the elongated grains is apparently a prior flake boundary and was formed during the hot pressing operation.

Fracture Toughness of Commercial Magnets- Conclusion

Application of a chevron notch bend test used for brittle ceramics and some intermetallic alloys to measure fracture toughness was applied to $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnets. A series of fracture toughness measurements on commercially produced $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnets showed consistent differences between 8 sets of specimens from four different manufacturers utilizing two different production methods. Fracture toughnesses ranged from 2.5 to 5.5

$\text{MPa}\sqrt{\text{m}}$. These baseline measurements are an essential first step to improving toughness utilizing techniques similar to those employed in some ceramics and in some intermetallic alloys.

Fig. 3. Fracture surface of a Magnequench MQ3-E magnet.

Fig. 4. Fracture surfaces of (a) Vaccumschmelze 42, (b) Sumitomo 40, and (c) Shin-Etsu 45 magnets.

Process Variables and Alloy Modifications-Summary

The effect that process variables have on fracture toughness of hot pressed magnets was studied. These variables included press pressure, press time, press direction, middle vs near end of a pressed magnet, powder size, and post specimen anneals for stress relief. Differences of up to $0.5 \text{ MPa}\sqrt{\text{m}}$ were observed. The higher values occurred for the production values. It was reassuring that the toughness test measurement on only 5 specimens for each condition was sufficiently sensitive to show such agreement with the empirical production floor experience. Having a reliable test that could track small changes would then allow small changes in toughness to be correlated with changes that were made to alloy compositions. Alloy modifications were first made by blending in powder additives with a limit of 2 vol%. The limit of 2% was established so as to not adversely affect the magnetic properties. Toughness and magnetic properties were measured and correlated with fracture surface images. Modifications to the base alloy compositions were also made. Compositions were selected based on expected amount of grain boundary phase and some compositions were selected based on analysis of the grain boundary phase in the toughest of the cast commercial magnets.

Process Variables and Alloy Modifications- Experimental Procedure

New compositions were made both by powder blending and by alloy modification before melt spinning. All final magnets were hot pressed as MQ2 magnets following Magnequench's standard procedure unless otherwise noted. Fracture toughness testing was performed on $5 \times 5 \times 40 \text{ mm}$ specimens with a chevron notch as described previously. Chemical analysis was performed at Magnequench International by Induction Coupled Plasma Spectroscopy, ICP. Magnetic properties were measured at Magnequench in a hysteresisgraph on previously pulsed magnetized specimens. Fracture surfaces were observed in a field emission gun equipped SEM with an energy dispersive spectrometer that was used for chemical analysis.

Process Variables and Alloy Modifications-Results

Table 2 shows fracture toughness as a function of press time, press pressure, specimen position and after a stress relieving post press anneal. Each value is an average of usually 5 tests with a typical standard deviation of $\pm 0.2 \text{ MPa}\sqrt{\text{m}}$. More detailed results including magnetic properties and chemistries are presented in the appendix. E1 and H are standard Magnequench grades of melt spun powder, while 450 was an experimental composition. The typical manufacturing condition is a 20 s press at 7 tons/ in² (96 MPa). The original

manufacturing conditions were determined by optimizing the magnetic properties and producing a consolidated hot pressed magnet that held together. For example, 7 tsi press pressure is better than 5 but a higher pressure of 11 tsi is of little further benefit. Likewise, longer press times also showed no benefit. However, post press anneals resulted in a rapid drop in toughness correlating with excessive grain growth of just a few grains, as shown in Fig. 5.

A number of different powder additives were blended into E1 grade powder and some E1 powder was CVD coated with Fe. These results are presented in Table 3. Further details are in the appendix. Of these additives, copper and brass were the most beneficial. Subsequent tests concentrated on copper additions by varying the volume fraction, press pressure and powder size. Differences of up to 0.5 MPa√m were obtained depending on the variable.

The commercial magnet with the highest fracture toughness measured was a Vacuumschmelze 42 magnet. This magnet was made by the cast, crush and sinter technique yielding a large grained material. In these large grain magnets, a greater fraction of the low melting point eutectic composition intergranular phase forms. This particular alloy, as shown in Fig. 6, with the highest toughness also had the most ductile appearing grain boundary phase. Since EDS analysis of this phase showed an aluminum enrichment, a series of alloys were melt spun

Table 2 Fracture toughness as a function of press time, press pressure, specimen position, and post press anneal

Grade	variable	MPa√m
450	20s press	2.7
450	60s press	2.4
450	120s press	2.6
E1	20s press	2.5
E1	60s press	2.9
E1	120s press	3.3
E1	5 tsi	1.9
E1	7 tsi	2.5
E1	11 tsi	2.6
MQ3	middle	2.8
MQ3	end	2.5
H	20s press	1.9
H	60s press	2.0
H	150s press	2.1
H	400s press	2.2
H	1 h 500° C	1.6
H	10 500° C	1.3
H	100h 500° C	1.3

Fig. 5. H grade hot pressed magnet after an additional 100 h anneal at 500°C.

with higher aluminum levels and the results shown in Table 4. Based on the improvements noted with the blending of copper powders, copper additions were also made to the base composition before melt spinning. Unfortunately, neither modification improved the toughness.

A correlation was noted from previously described tests (repeated as the first three

entries in Table 5) between toughness and the expected fraction of grain boundary phase based on a mass balance between the major magnetic phase, $\text{Nd}_2\text{Fe}_{14}\text{B}$, the eutectic grain boundary phase Nd_7Fe_3 and the phase formed from the rejected boron, $\text{Nd}_2\text{Fe}_7\text{B}_6$. Toughness

Table 3 Toughness with blended powder additives

Powder Grade plus additive	other	Toughness MPa $\sqrt{\text{m}}$
E1		2.5
E1+ Fe		2.6
E1 + Sn		2.6
E1 + Mo		2.5
E1 + CaF_2		2.4
E1 + Cu		3.4
E1 + Nd_2O_3		2.7
E1 + Zn		2.6
E1 + Al		2.6
E1 + Brass		3.6
450		2.7
E1 + Zirconia		2.8
E1 + NiTi		2.6
E1 + Ni_3Al		2.2
E1 + Au		2.9
E1 with CVD Fe		2.2
E1 + 0.5% Cu		2.5
E1+ 1% Cu		2.4
E1 + 2% Cu		2.9
E1 + 4% Cu		2.9
E1 + 2% Cu	5 tsi	2.2
E1 + 2% Cu	7 tsi	2.9
E1 + 2% Cu	11 tsi	3.0
E1 + 2% Cu	<5 μm	2.8
E1 + 2% Cu	<325 mesh	2.8
E1 +2% Cu,	<200, >325	2.2
E1 +2% Cu	>200	2.2

Fig. 6 Fracture surface of a large grained Vacuum-schmelze 42.

Table 4 Toughness of compositions modified before melt spinning

Composition	MPa $\sqrt{\text{m}}$
H	2.4
H + 0.13 wt.% Al	2.4
H + 0.27% Al	2.2
H + 0.61% Cu	2.1
H + 0.5% Cu	1.9

was found to increase from 2.5 to 3.5 as the percent grain boundary phase increased from 7 to 11%. The 450 is a ShinEtsu composition that was melt spun by Magnequench and the specimen labeled 273 was a Vacuumschmelze composition also melt spun by Magnequench. Both these compositions are from the cast and crush processing route and so typically are higher in rare earth to produce more of the grain boundary eutectic phase necessary for the sintering operation. The melt spun material of Magnequench is closer to stoichiometry. As can be seen in the last 5 entries, this correlation did not continue to hold.

Alloy oxygen levels were changed by varying the starting powder size. Environmental effects as happens in reactive aluminides with water vapor was also tested for by comparing tests done in air, vacuum, and under oxygen. Neither set of experiments showed an effect, see Appendix 1 for details.

Process Variables and Alloy Modifications-Conclusions

Within the severe constrain of no more than 2 volume percent nonmagnetic additions, no significant improvement, greater than 0.5 MPa√m, to the toughness was produced. In other brittle intermetallics, ductile phase toughening often requires 25 to 50% to produce the desired benefits. The large grained sized material made by the cast, crush, and sinter processing method have an inherent advantage in toughness, due both to larger grains and due a greater volume fraction of metallic grain boundary phase. However, this greater volume of metallic grain boundary phase makes these alloys more susceptible to corrosion. At this point in this CRADA project, it was decided to emphasize the more fundamental aspects of the fracture process.

Table 5 Correlation of toughness with expected amount of grain boundary phase*

Grade		Total Rare Earth	B level	other	Toughness, Mpa√m	Expected GB phase, %
E1	Magnequench	30.9%	0.94%	0.1% Al	2.5	7
450	ShinEtsu	32.0	1.0	0.24 Al	2.7	8.6
H	Magnequench	31.2	0.89		3.5	11
267	should be lower toughness	29.9	0.97		2.0	5
268	should be higher	29.4	0.87		2.2	13
266	should be higher	31.9	0.81		2.0	19
273	Vacuumschmelze	32.2	1.01	0.4 Al	1.9	9
465	should be higher	34.3	0.98	0.3 Al	2.2	12

*compositions in weight percent

Fracture Mode-Summary [12]

Efforts to understand and improve the fracture toughness of $\text{Nd}_2\text{Fe}_{14}\text{B}$ permanent magnets require an understanding of the fracture process itself. Cleavage plane orientations in $\text{Nd}_2\text{Fe}_{14}\text{B}$ were identified by X-ray diffraction and found to be rather random. Cleavage fracture surfaces often exhibited smooth curvatures with no evidence for cleavage steps. The small grain sizes of less than 100 nm in Magnequench MQ material preclude an easy assessment of the fracture mode by scanning electron microscopy. Auger electron spectroscopy showed that much of the surface is covered with a 1 nm thick layer of a neodymium-rich phase, presumably the 70Nd-30Fe eutectic phase, suggesting that the hard $\text{Nd}_2\text{Fe}_{14}\text{B}$ grains do not cleave but instead failure is at or in the grain boundary phase.

Fracture Mode- Introduction

Efforts to understand and improve the fracture toughness of $\text{Nd}_2\text{Fe}_{14}\text{B}$ permanent magnets require an understanding of the fracture process itself. It has been known that $\text{Nd}_2\text{Fe}_{14}\text{B}$ has no deformation modes and therefore is completely brittle. Earlier studies have measured toughness and strength but have not addressed the fracture process [1-3,13]. Generally, low symmetry, large-unit-cell intermetallics fail by cleavage. Even at hot pressing temperatures of 750°C, when the low melting point eutectic phase forms and serves as a sintering aid, no slip deformation is thought to occur. Cleavage during this step in processing may aid development of anisotropy much as slip does in texture development in many metal alloys. Because of the small grain sizes (less than 100 nm) in the melt spun Magnequench MQ magnets, it is difficult to conclude much about the fracture process based on images from a scanning electron microscope (SEM) of the fracture surface.

In this work, orientations of cleavage faces formed from fracture of single crystals of $\text{Nd}_2\text{Fe}_{14}\text{B}$ were identified by x-ray diffraction (XRD). Auger electron spectroscopy (AES) analysis was used to measure the composition of the fracture surface as a function of depth in order to confirm the presence of the expected grain boundary phases.

Fracture Mode- Experimental Procedure

Single crystals, up to $10 \times 10 \times 2$ mm, of $\text{Nd}_2\text{Fe}_{14}\text{B}$ were grown at Ames Laboratory by the slow cooling of a Nd-rich ternary melt and then were crushed to obtain cleavage faces. XRD orientations of crystals with fairly flat cleavage faces were made using a 4-circle x-ray diffractometer. After orientation in the goniometer, reflected laser light was used to measure the angular curvature of the face. Stereo images were made of these cleavage faces in a field emission SEM. Surface imaging was also performed in the contact mode in

Fig. 7. SEM micrographs of fracture surfaces in a MQII magnet showing (a) the small grain size and difficulty in assessing the fracture mode and (b) a similar sample after a 4

a Park Scientific AutoProbe XL atomic force microscope (AFM). To measure grain boundary compositions, a series of alloys, all processed by the normal MQII route, were analyzed by AES. After specimen insertion and bake out, the specimens were fractured under UHV conditions inside the spectrometer immediately before analysis. The AES data were collected using a PHI Model 590 Scanning Auger Microprobe with a beam energy of 5 keV and a current of ~140 nA. The data were collected in a voltage to frequency conversion mode.

Fracture Mode-Results

Figure 7a shows a fracture surface of a Magnequench MQII magnet. The grain size is generally less than 100 nm and much too small to determine orientations. Figure 7b shows a similar specimen after an anneal for 4 h at 800°C. Attempts were made to orient the larger faces using electron backscattered patterns (EBSP) also called backscattered Kikuchi patterns (BKP) in a SEM. However, due to the low symmetry, large unit cell and the low incidence angle (the specimen was tilted nearly parallel to the incident electron beam) and the roughness of the fracture surface, analyzable images could not be obtained.

Table 6 XRD Determined Cleavage Plane Normals

Area #	Cleavage Plane Normals		
	h	k	l
1	4.0	0.4	0.7
2	4.0	0.6	0.5
3	1.5	1.4	4.8
5	3.2	0.7	3.4
7	2.4	1.9	3.7
8	2.8	0.7	2.6

The orientations of cleavage plane normals measured on single crystals are listed in

Fig. 8. Stereographic triangle with marked cleavage face orientations as measured by XRD. Faces labelled 1, 2, and 5 are drawn as lines since they were curved in one direction. The length of the line represents the angular range. The areas labelled 3, 7, and 8 were hemispherical and the circle covers the angular range of the cleavage faces.

Table 6 and shown graphically on a stereographic triangle in Fig. 8. The circles in Fig. 8 represent the approximate angular range of the hemispherical faces oriented by XRD. The lines in Fig. 8 represent the approximate cylindrical angular range of the other faces analyzed. Note that $\text{Nd}_2\text{Fe}_{14}\text{B}$ has a tetragonal unit cell and so the stereographic triangle shown consists of what looks like two regular cubic stereographic triangles. Even though these orientations appear in a band, they cover such a large fraction of the stereographic projection that they therefore are quite random.

Often, curved cleavage fracture surfaces in intermetallic alloys are actually stepped.

Fig. 9. SEM micrographs of cleavage fracture surfaces in $\text{Nd}_2\text{Fe}_{14}\text{B}$ single crystals. Flat fracture surfaces were oriented by XRD. The top of figure 9a is looking obliquely at an almost flat region analyzed as area 1, while the lower half of the figure is a chipped edge showing the smoothly curved fracture surfaces. Figure 9b is looking down on a relatively flat region analyzed as area 2.

Fig. 10. AFM image of surface of cleavage face of area #3. No small steps were imaged.

Stereo SEM micrographs of specimens in this study showed curved faces with no evidence of stepping. Figure 9 shows two SEM micrographs of two flat areas that were oriented by XRD and had adjacent chipped areas with highly curved surfaces. AFM scans, Fig. 10, also indicated that curved surfaces were present. Cleavage in intermetallic alloys is often planar with particularly “weak” planes favored. In tetragonal structures, the c-face is often the cleavage plane. The unit cell of $\text{Nd}_2\text{Fe}_{14}\text{B}$ is a layered structure with a mixed layer on the c plane followed by two iron-only layers. Because of this structure, cleavage on (001) was expected. However, no calculations of weak bonding directions have been performed on this structure as has been done on several other intermetallic alloys. The lack of preferred cleavage planes and the curved nature observed here suggest a fracture similar to that expected in amorphous or glassy materials.

The $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnets consist of a mechanically-hard, nondeforming boride phase sintered together with a metallic Nd-rich phase at the grain boundaries. Transmission electron microscopy (TEM) images of the MQII material show very little grain boundary phase mainly due to the near stoichiometric composition used. Detailed TEM analyses of these materials have described the presence of several grain boundary phases from 1 to 10 nm thick [15-16]. The thinner areas were found to have a composition of NdFe_3 , the thicker areas were neodymium rich and 70Nd-30Fe was present at grain boundary junctions. However, these results covered a limited number of boundaries and areas analyzed. AES was used first to help confirm that the fracture surface was intergranular and secondly to gain an estimate of the area fraction covered by grain boundary phase and a confirmation to the composition. So far, similar analysis by atom probe has failed on bulk MQII material due to the difficulty in specimen preparation.

Figure 11 shows AES data for 5 alloys analyzed, all processed similarly. The data

Fig. 11. Averaged AES scans for neodymium and boron levels as a function of depth for the 5 alloys listed in Table 7. The surface composition was close to that of the low melting point eutectic 70Nd-30Fe phase suggesting that a substantial fraction of the fracture surface was covered with a 1 nm thick layer of the eutectic phase.

plotted are averages of more than 5 scans on each alloy. The curve labelled 10 is a normal E grade MQII magnet. Base compositions are given in Table 7. Note, the boron compositions after 30 mins (~30 nm removed) of argon sputtering from the lowest data point on the graph to the highest are in the correct order, 0.81, 0.87, 0.89, 0.97, and 1.01 wt.%, suggesting that the measurement technique is relatively accurate.

Table 7 Compositions of alloys analyzed by AES.

Alloy #	RE level		B level	
	wt.%	at.%	wt.%	at.%
75	29.4	13.3	0.87	5.3
74	29.9	13.5	0.97	5.9
10	30.9	14.1	0.89	5.4
73	31.9	14.7	0.81	5.0
71	32.2	14.8	1.01	6.2

The neodymium concentration after 30 m of sputtering are almost in order. From the lowest to highest curve on the graph, the data points are from alloys with 29.4, 29.9, 30.9, 32.2 and 31.9 wt.% total rare earth. However, the absolute bulk composition from the AES analysis is high. The surface concentration of neodymium is more than twice the bulk concentration. The iron concentration curves, not shown, are opposite that of the neodymium. The neodymium enrichment levels observed suggest that up to 3/4 of the surface is coated with a 1 nm thick layer of the 70Nd-30Fe phase. After 3 min of sputtering (3 nm removed) bulk compositions are reached.

Fracture Mode- Conclusions

Fracture in the small-grained $\text{Nd}_2\text{Fe}_{14}\text{B}$ MQII magnets is intergranular with a 1 nm thick layer of the eutectic 70Nd-30Fe phase over possibly 3/4 of the fracture surface. While brittle intermetallics usually cleave on preferred planes, $\text{Nd}_2\text{Fe}_{14}\text{B}$ appears to be non-directional when cleaving.

High Energy Product Ribbons-Introduction [17]

The bonded Nd-Fe-B market has experienced the fastest growth of any permanent magnet market [18]. Rapidly solidified Nd-Fe-B forms the basis for this bonded magnet industry. Rapid solidification is carried out by melt spinning, producing a highly stable and magnetically hard microstructure. The melt spun ribbons are ground into powder before being processed into bonded magnets. Of the various isotropic powders manufactured by Magnequench International (MQI), MQP-B powder has become the industry standard for multipole rings used in stepper and spindle motors [19]. The continuous process and product improvements led to the development of a new powder, MQP-B⁺. The B_r value of this powder, >8.4 kG, is higher than MQP-B and the new powder has an improved squareness of the demagnetization curve with a minimum intrinsic induction B_d equal to 6 kG at a reverse field of 6kOe as compared to 4.5 kG for the original MQP-B powder. The magnetic properties are given in Table 8. A typical demagnetiza-

Table 8 Magnetic Properties of the Analyzed Ribbons

	No Demag Factor	With 0.333 Demag Factor
<u>Experimental Values</u>		
B_r , kG	8.5	9.2
H_{ci} , kOe	10.0	10.0
BH_{max} , MGOe	14.7	16.5
<u>Specification Values</u>		
B_r , kG	>8.4	
H_{ci} , kOe	9.0 to 10.5	
B_d (min), kG	6.0 (reverse field of 6 kOe)	

tion curve is shown in Fig. 12. The specification values for the commercial version of MQP-B⁺ are also given in Table 8. Note that these values are given without any demag factor applied as is normally done by Magnequench for all their isotropic powders.

Fig. 12. BH curve for MQP-B⁺.

This study focuses on a microstructural analysis of this melt spun ribbon using transmission and scanning electron microscopy (TEM and SEM), atom probe field ion microscopy (APFIM) and Au-

ger electron spectroscopy (AES). Atom probe and AES are relatively unused techniques in the study of the microstructure of magnetic materials and were used here to gain some information on the compositions and thickness of any grain boundary phase. AES has the advantage of simple specimen preparation, the specimen is broken inside the UHV analysis chamber and immediately analyzed. Previous studies have shown that the fracture even in the small grained Magnequench material is intergranular [12]. The atom probe has the advantage of a relatively direct and therefore accurate compositional measurement of very small phases. The small grain sizes, 30 to 40 nm, present in the melt spun ribbons and presumably thin grain boundary phases are a good match to the scale examined by APFIM [20].

High Energy Product Ribbons- Experimental Procedure

Pieces of B⁺ ribbon, approximately $5 \times 2 \times 0.04$ mm thick were supplied by Magnequench. Some of the ribbons analyzed by atom probe were melt spun at ORNL from an ingot supplied by Magnequench. These ribbons were 0.02×0.4 mm in cross section and made specimen preparation for the atom probe easier. The nominal composition of the B⁺ ribbons was Fe-5 Co-0.9 B-28-total rare earth (wt.%). Specimens for TEM were made by ion milling in both the normal plan view orientation and in the cross section orientation. The plan view (top view) specimens that were made by back thinning to either the wheel side or the free side did not work due to the surface roughness. Cross section specimens (side view) were made by the usual techniques involving sandwiching the ribbons between

layers of copper sheet, cutting out and mechanically thinning 3 mm disks following by mechanical dimpling and ion milling. Specimens for the atom probe were made following usual techniques in a 5% perchloric-acetic acid mixture. The analysis was done in ORNL's energy-compensated atom probe at a specimen temperature of 50 K and a pulse fraction of 15%. Specimens for AES were introduced into the high vacuum system and fractured under UHV conditions inside the PHI model 590 Scanning Auger Microprobe spectrometer immediately prior to analysis. Analysis was done at 5 keV and a current of ~140 nA. The data were collected in a voltage to frequency conversion mode.

High Energy Product Ribbons-Results

SEM, Fig. 13, and TEM, Fig. 14, images showed similar grain sizes and shapes throughout the thickness of the ribbon. The SEM images showed a recently fractured edge. The grain size was uniform throughout the thickness of the specimen. TEM images

Fig. 13. SEM micrographs of melt spun ribbon, MQI-B⁺. (a) and (b) show rough and smooth (wheel) sides, respectively, while (c) is a fractured edge near the rough side and (d) is a fractured edge from near the wheel side.

Fig.14. TEM micrographs of MQI-B⁺ powder showing uniformity of grain size through the thickness of the melt spun ribbon. Cross section images are of near one surface (a), near the middle (b), and near the other surface (c). (d) is a plan view from near the middle of the ribbon. All images are at the same magnification.

also showed an isotropic structure with similar grain sizes throughout the ribbon. Figures 14a, b, and c show cross section views (sideways) while (d) is a plan view, (looking down on the ribbon).

Preliminary results from the atom probe analysis of ribbon that was melt spun at ORNL showed compositions very close to the compositional analysis of the starting ingot, as shown in Table 9. For this analysis, 30,000 ions were collected essentially from a cylinder of material approximately 2 nm in diam and 500 nm tall. Analysis of the MQI-B⁺ powder gave similar results. Both analyses showed higher than expected carbon levels. Due to the low-symmetry, large, unit-cell structure, FIM images are more complex than typical metals making identification of grain boundaries and grain boundary phases more difficult (Fig. 15). As shown in Fig. 16, fluctuations of Nd-Fe ratio were observed in the composition profiles. One shown in Fig. 16 reached nearly 35% Nd. However, as a thin grain boundary phase sweeps past the probe hole, the atom probe analyzes both the potential grain boundary phase and some matrix.

AES analysis was performed because it is a relatively easy technique for accurately depth profiling an area. Previous results by AES (3) have shown that the fracture surfaces of MQ2 magnets are indeed intergranular with approximately a

1 nm thick layer of the expected 70Nd-30Fe phase present over most of the area of the fracture surface. Specimens of the ribbon were mounted in a similar way in the AES and

Table 9 Atom probe analysis of grain interiors (at.%)

	MQI-B ⁺ Powder		ORNL melt spinning	
	Atom Probe	nominal	Atom Probe	Original Ingot
Fe	75.3±0.5	76.6	77.5±0.2	77.3
Nd*	12.9±0.4	12.6	11.2±0.2	13.6
B	5.2±0.2	5.5	6.6±0.14	6.1
Co	5.5±0.2	5.4	3.3±0.1	2.7
C	1.2±0.1	-	0.5±0.04	NA
Al	0.14±0.04	-	0.3±0.03	0.27
Si	0.11±0.04	-	0.6±0.04	NA
Mn	0	-	0	0.04
Ga	0	-	0	0.04

*Nd analysis includes other REs, NA- not analyzed, compositions in at.%

Fig. 15. APFIM image of Nd₂Fe₁₄B melt spun ribbon. Due to the low symmetry and large unit cell, distinct rings and layers are absent.

Fig. 16. Composition profile made by APFIM on a B⁺ ribbon through a probe.

were broken in the analysis chamber. Analysis of the ribbon also showed a similar neodymium enrichment at the surface. Figure 17 shows plots as a function of depth for Nd, Fe, B, and Co. After an analysis was performed, the surface was subjected to an argon ion sputtering, with a sputtering rate of approximately 1 nm per minute. The results here indicate that a fracture surface enrichment similar to that of bulk MQ2 magnets was present in the ribbon.

High Energy Product Ribbons-Conclusions

The new B⁺ ribbons showed a uniform grain size and shape distribution through the thickness of the melt spun ribbon. Grain sizes ranged from 20 to 40 nm. AES showed neodymium enrichment on a fracture surface and corresponding iron and boron depletion suggesting that the eutectic 70Nd-30Fe phase is present with a thickness of approximately 1 nm. Atom probe composition analysis of grain interiors gave results very close to the nominal composition and some preliminary evidence of a grain boundary phase.

Fig. 17. AES spectra for Nd, Fe, Co, and B for a specimen of B* powder fractured in the analysis chamber. The argon ion sputtering rate was approximately 1 nm per minute.

Summary

It is clear comparing the Magnequench melt spun material to the large grained crushed and sintered material, that grain size is a major factor in determining the fracture toughness. The chevron notch test was found with just 4 or 5 tests, to produce consistent and believable toughness measurements, based on production experience with process variables. Unfortunately additions limited to 2% was not enough to result in any ductile phase reinforcement. Fracture in the Magnequench magnets was intergranular, while fracture in the large grained material was transgranular cleavage. Contrary to many intermetallics, cleavage facets were not on preferred planes but curved with a random orientation. In the case of the small grained Magnequench material, the grains of hard $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase tended not to break but instead deformation and fracture were concentrated in the weaker intergranular phases.

INVENTIONS

None

COMMERCIALIZATION POSSIBILITIES

None

PLANS FOR FUTURE COLLABORATION

This study could be extended to sintered material that is inherently tougher because of larger grains. If a grade of neodymium permanent magnets was sold based on a combination of toughness and magnetic properties it would probably be based on a larger grained material. A couple of unsuccessful attempts have been made to secure support for such an investigation.

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Appendix 1 Experimental Data

file name	Magnequench #	description of material	major variable	Toughness Values		Magnetic Properties		Composition of melt spun magnet material before any powder blending, wt. %														
				toughness-ave., MPa _y m	fracture w controlled crack	Remanance, B, KG	Coercivity, H _c KOe	Energy Product, MGOe	Iron	TRE	Boron	Cobalt	Oxygen	Nitrogen	Manganese	Magnesium	Gallium	Aluminum	Calcium	Copper	Zirconium	other
ndma7			E1	2.4 ± 0.3																		
ndma8			MC3	3.1 ± 0.3																		
ndma9			MC3	2.7 ± 0.2																		
ndma10			Sumilomo	4.3 ± 0.2																		
ndma11			commercial material	5.5 ± 0.2																		
ndma12			commercial material	4.9 ± 0.6																		
ndma13			commercial material	2.6 ± 0.1																		
ndma14			ShinEtlau	2.7 ± 0.1																		
ndma15			E1 trade	2.7 ± 0.1																		
ndma16			Fe powder blended	2.7 ± 0.1																		
ndma17			Sn powder blended	2.7 ± 0.1																		
ndma18			Mo powder blended	2.7 ± 0.1																		
ndma19			Cu powder blended	2.7 ± 0.1																		
ndma20			CaF ₂ powder blended	2.7 ± 0.1																		
ndma21			Cu powder blended	2.7 ± 0.1																		
ndma22			Nd ₂ O ₃ powder blended	2.7 ± 0.1																		
ndma23			Nd ₂ O ₃ powder blended	2.7 ± 0.1																		
ndma24			Zn powder blended	2.7 ± 0.1																		
ndma25			Al powder blended	2.7 ± 0.1																		
ndma26			brass powder blended	2.7 ± 0.1																		
ndma27			ShinEtlau composition melt spun	2.7 ± 0.1																		
ndma28			Diado extrusion	2.7 ± 0.1																		
ndma29			H	2.7 ± 0.1																		
ndma30			9308-01	2.7 ± 0.1																		
ndma31			9308-02	2.7 ± 0.1																		
ndma32			9308-03	2.7 ± 0.1																		
ndma33			9308-04	2.7 ± 0.1																		
ndma34			9308-05	2.7 ± 0.1																		
ndma35			9308-06	2.7 ± 0.1																		
ndma36			9308-07	2.7 ± 0.1																		
ndma37			9308-08	2.7 ± 0.1																		
ndma38			9308-09	2.7 ± 0.1																		
ndma39			9308-10	2.7 ± 0.1																		
ndma40			9308-11	2.7 ± 0.1																		
ndma41			9308-12	2.7 ± 0.1																		
ndma42			9308-13	2.7 ± 0.1																		
ndma43			9308-14	2.7 ± 0.1																		
ndma44			9308-15	2.7 ± 0.1																		
ndma45			9308-16	2.7 ± 0.1																		
ndma46			9308-17	2.7 ± 0.1																		
ndma47			9308-18	2.7 ± 0.1																		
ndma48			9308-19	2.7 ± 0.1																		
ndma49			9308-20	2.7 ± 0.1																		
ndma50			9308-21	2.7 ± 0.1																		
ndma51			9308-22	2.7 ± 0.1																		
ndma52			9308-23	2.7 ± 0.1																		
ndma53			9308-24	2.7 ± 0.1																		
ndma54			9308-25	2.7 ± 0.1																		
ndma55			9308-26	2.7 ± 0.1																		
ndma56			9308-27	2.7 ± 0.1																		
ndma57			9308-28	2.7 ± 0.1																		
ndma58			9308-29	2.7 ± 0.1																		
ndma59			9308-30	2.7 ± 0.1																		
ndma60			9308-31	2.7 ± 0.1																		
ndma61			9308-32	2.7 ± 0.1																		
ndma62			9308-33	2.7 ± 0.1																		
ndma63			9308-34	2.7 ± 0.1																		
ndma64			9308-35	2.7 ± 0.1																		
ndma65			9308-36	2.7 ± 0.1																		
ndma66			9308-37	2.7 ± 0.1																		
ndma67			9308-38	2.7 ± 0.1																		
ndma68			9308-39	2.7 ± 0.1																		
ndma69			9308-40	2.7 ± 0.1																		
ndma70			9308-41	2.7 ± 0.1																		
ndma71			9308-42	2.7 ± 0.1																		
ndma72			9308-43	2.7 ± 0.1																		
ndma73			9308-44	2.7 ± 0.1																		
ndma74			9308-45	2.7 ± 0.1																		
ndma75			9308-46	2.7 ± 0.1																		
ndma76			9308-47	2.7 ± 0.1																		
ndma77			9308-48	2.7 ± 0.1																		
ndma78			9308-49	2.7 ± 0.1																		
ndma79			9308-50	2.7 ± 0.1																		
ndma80			9308-51	2.7 ± 0.1																		
ndma81			9308-52	2.7 ± 0.1																		
ndma82			9308-53	2.7 ± 0.1																		
ndma83			9308-54	2.7 ± 0.1																		
ndma84			9308-55	2.7 ± 0.1																		
ndma85			9308-56	2.7 ± 0.1																		
ndma86			9308-57	2.7 ± 0.1																		
ndma87			9308-58	2.7 ± 0.1																		
ndma88			9308-59	2.7 ± 0.1																		
ndma89			9308-60	2.7 ± 0.1																		
ndma90			9308-61	2.7 ± 0.1																		
ndma91			9308-62	2.7 ± 0.1																		
ndma92			9308-63	2.7 ± 0.1																		
ndma93			9308-64	2.7 ± 0.1																		
ndma94			9308-65	2.7 ± 0.1																		
ndma95			9308-66	2.7 ± 0.1																		
ndma96			9308-67	2.7 ± 0.1																		
ndma97			9308-68	2.7 ± 0.1																		
ndma98			9308-69	2.7 ± 0.1																		
ndma99			9308-70	2.7 ± 0.1																		
ndma100			9308-71	2.7 ± 0.1																		
ndma101			9308-72	2.7 ± 0.1																		
ndma102			9308-73	2.7 ± 0.1																		
ndma103			9308-74	2.7 ± 0.1																		
ndma104			9308-75	2.7 ± 0.1																		
ndma105			9308-76	2.7 ± 0.1																		
ndma106			9308-77	2.7 ± 0																		

Appendix 1 Experimental Data

file name	Magnequench #	description of material	major variable	Toughness Values			Magnetic Properties			Composition of melt spun magnet material before any powder blending, wt. %															
				toughness-ave., MPa/m	toughness-stn. dev	fraction w controlled crack growth	Remanence, B _r KG	Coercivity, H _c KOe	Energy Product, MGOe	Iron	TRE	Boron	Cobalt	Oxygen	Nitrogen	Manganese	Magnesium	Gallium	Aluminum	Calcium	Copper	Zirconium	other		
alInco		TDK	commercial material	13.3 ±1.0	yes																				
ndmq54		H, 20s press		2.1 ±0.3	0/5		8	-19.8	14.6	bal	-31.25	-0.89													
ndmq55		H, 20s, 60s @ 750°C	grain size	1.9 ±0.3	2/4		8.2	-19.1	15.4	bal	-31.25	-0.89													
ndmq56		H, 20s, 150s @ 750°C	grain size	1.9 ±0.2	1/4		8.2	-2.1	15.6	bal	-31.25	-0.89													
ndmq57		H, 20s, 400s @ 750°C	grain size	2.2 ±0.1	2/2		8.1	-19.8	14.6	bal	-31.25	-0.89													
ndmq58		H, 60s press	press time	1.9 ±0.1	3/4		8	-20.4	14.6	bal	-31.25	-0.89													
ndmq59		H, 150s press	press time	2.2 ±0.1	4/4		8	-1.7	13.6	bal	-31.25	-0.89													
ndmq60		H, 400s press	press time	2.2 ±0.2	4/4		8	-19.4	13.6	bal	-31.25	-0.89													
ndmq61		H, 20s, 1 hr 500C vac	stress relief, grain size	1.7 ±0.2	1/2					bal	-31.25	-0.89													
ndmq62		H, 20s, 10 hr 500C vac	stress relief, grain size	1.3 ±0.0	3/3					bal	-31.25	-0.89													
ndmq63		H, 20s, 100 hr 500C vac	stress relief, grain size	1.3 ±0.4	3/3					bal	-31.25	-0.89													
ndmq64		CVD Fe on F powder, #1		2.2 ±0.2	2/2		8	-17.9	14.0																
ndmq65		CVD Fe on F powder, #1		2.2 ±0.1	4/4		7.6	-16.8	12.9																
ndmq66	263	H + 0.27 Al then melt spun		2.2 ±0.3	1/5					61.52	30.74	0.825	6.07			0	0	0.59	0.27	0					
ndmq67	262	H + 0.13 Al then melt spun		2.4 ±0.7	2/5					61.41	30.87	0.84	6.16			0	0	0.58	0.13	0					
ndmq68	274	H + 0.61 Al then melt spun		2.1 ±0.3	1/3					60.44	31.29	0.84	6.19			0	0	0.63	0.61	0.001					
ndmq69	264	H + 0.26 Cu then melt spun		2.1 ±0.5	2/4					61.41	31.14	0.82	6.03			0	0	0.57	0.03	0	0.26				
ndmq70	265	H + 0.5 Cu then melt spun		1.9 ±0.2	3/3					61.49	30.86	0.84	6.19			0	0	0.59	0.03	0	0.5				
ndmq71	273	Vacuumschmelze compo then melt spun		1.9 ±0.1	0/5					65.26	32.23	1.01	1.07			0	0	0.02	0.4	0.001					
ndmq72	465	Vac mistake composition		2.2 ±0.3	2/5					63.26	34.26	0.98	1.13	0.015	0.002	0.02	0.001	0.026	0.295	0	0				
ndmq73	266A	31.9 TRE		2.0 ±0.2	5/5					60.49	31.94	0.81	6.19			0	0	0.55	0.03	0					
ndmq74	267A	29.9 TRE		2.0 ±0.2	4/5					62.42	29.92	0.97	6.13			0	0	0.54	0.03	0					
ndmq75	268A			2.3 ±0.3	2/5					62.91	29.42	0.87	6.23			0	0	0.55	0.026	0					
ndmq76		H polished, 3 pt bend in MPa	yield stress in MPa=	163.4 ±31.8																					
ndmq77		H as ground, 3 pt bend	yield stress in MPa=	132.6 ±19.7																					
o2nd78		H	tested in oxygen	1.8 ±0.3	no																				
o2s142-1		Vacuumschmelze 42	tested in oxygen	5.5 #####	yes																				
o2s142-2		Vacuumschmelze 42	tested in water	5.4 #####	yes																				
o2s142-3		Vacuumschmelze 42	tested in vacuum	7.2 #####	yes																				
n5-6		HDDR, Nd16Fe76B8	250 MPa green then 800°C press at 200 MPa	3.4 ±0.2	3/5		7.2	12.5	10.6	76 at.%	16 at.%	8 at.%													
n5-8		HDDR, Nd16Fe76B8	210 MPa green then 800°C press at 200 MPa	3.1 ±0.3	3/5																				
n11-8		HDDR, Nd14Ba79B8	20 s press at 830°C	3.2 ±0.3	3/5		7.6	10	12.0	79 at.%	14 at.%	7 at.%													
n11-9		HDDR, Nd14Ba79B8	40 s press at 830°C	3.3 ±0.2	3/5																				
neocomp12		INEL 435-2, S<75		3.8 ±0.1	close		61	7.7	8.1	bal	34.8	1.08													
crumax45		from Crucible		4.2 ±0.2	yes																				
ndfe45		SHnEtsu	20 mm span length	4.2 ±0.3	yes																				
ndmq78		H grade	20 mm span length	2.4 #####	no																				
neocom11		my press F powder		2.4 ±0.1	no		7.2	17.6	11.2	bal	30.6	0.84	6.11	0.032	0.006	0.1		0.67	0.1						
neocom13		INEL 435-2, S50-75		5.7 ±0.1	yes		6.5	11.9	9.2	bal	34.8	1.08													
neocom14		INEL 435-2, S10-20		3.2 ±0.6	1/2		6.4	9.4	9.2	bal	34.8	1.08													
neocom15		INEL 465 (TiC), S<30		1.9 ±0.1	no					bal	30.1	1.1													
neocom16		INEL 465 (TiC), S<30, H800T10		2.0 #####	no					bal	30.1	1.1												3 Ti-3 C	
neocom17		INEL 465s10-20		#####			6.6	11.1	8.6	bal	30.1	1.1												3 Ti-3 C	
neocom18		INEL 465, (TiC), S10-20, H800T10		3.1 ±0.9	no					bal	30.1	1.1													
ndmq78		H, control specime	EDM hydrogen removal	2.4 #####	no					bal	-31.25	-0.89													
ndmq79		H, then 1 hr 325°C for H ₂ removal	EDM hydrogen removal	3.0 ±0.2	no					bal	-31.25	-0.89													
ndmq80	C2-1	H grade, >100 mesh, <35	powder size, oxygen level	2.3 ±0.3	1/3																				
ndmq81	C2-2	H grade, >325, <100	powder size, oxygen level	2.0 ±0.1	1/3																				
ndmq82	C2-3	H grade, <325	powder size, oxygen level	2.0 ±0.3	1/3																				
ndmq83	C2-4	H grade, mortar grinding	powder size, oxygen level	2.2 ±0.2	no																				
ndmq84	C2-5	H grade+0.5 wt% Cu, >325, <100	powder size, oxygen level	2.3 ±0.2	1/3																				

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