ABSTRACT

Garrell, Monika Gerda. Mechanical Properties of Injection-Molded Nd-Fe-B Type Permanent Magnets. (Under the direction of Dr. Albert Shih)

The goal of this research was to investigate the mechanical properties of injection molded Nylon and PPS-based Nd-Fe-B type magnets. The development of new Nd-Fe-B type magnetic materials and the advancement of near-net shape injection molding processes for magnetic component manufacturing have driven the needs to evaluate the mechanical properties of these newly developed materials. PPS (Polyphenylene-Sulfide) and Nylon (Polyamide) are the two most common binders used for these injection molded rare earth magnets.

Since magnetic materials are usually used at elevated and cryogenic temperatures in the automotive and computer industry, the temperature dependent properties ranging from -40 to 180 °C are critical for the design of devices utilizing permanent magnetic materials. To enlarge the use of bonded magnets, it is essential to establish a data-base of mechanical characteristics over the operational temperature range. This will provide valuable information for material designers to tailor the formulation and process parameters to achieve the desired mechanical properties.

This research included a series of mechanical properties testing following appropriate ASTM standards. Tensile and bending strengths were evaluated, since these are considered to be the most fundamental characteristics describing the mechanical behavior of materials. Young's modulus was measured using the dynamic impulse vibration method and compared with that obtained from tensile tests. Scanning Electron Microscopy (SEM) analyses indicated that the debonding on the Nd-Fe-B particle and Nylon interface was the main cause of failure at room and elevated temperatures.

MECHANICAL PROPERTIES OF INJECTION-MOLDED ND-FE-B TYPE PERMANENT MAGNETS

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BIOGRAPHY

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Table 4.1 Dynamic Modulus Comparison

NOMENCLATURE

- bwidth of flexural bar (mm)dapplied displacement (mm)
- f_f fundamental resonant frequency of bar in flexure in Hz
- \tilde{l} original gauge length of specimen (mm)
- ?l change in gauge length (mm)
- *m* mass of flexural bar in gramm (g)
- t thickness of flexural bar (mm)
- *x* horizontal coordinate of point of intersection of arcs (mm)
- *y* vertical coordinate of point of intersection of arcs (mm)
- *E* Young's Modulus (MPa or GPa)
- *K_f* Stress Concentration factor
- *L* length of flexural bar (mm)
- *R* Grindo-Sonic reading
- *RI* inner radius (mm)
- *RO* outer radius (mm)
- T_1 Correction factor
- *e* Strain (mm/mm or %)
- ? Angle describing arclength
- μ Poisson's Ratio
- s Stress (MPa)

1 INTRODUCTION

Permanent magnetic materials play an important part in powering accessories, emission control, and energy conversion in modern automobiles and trucks. In 2000, an average car employed approximately 25 electric motors, actuators, and sensors that contain permanent magnets. Some applications of the permanent magnets are as illustrated in Figure 1.1. Due to the high demand for better emissions control and more automation in the car, the average number of permanent magnet applications in a car is expected to rise to 32 by 2005.



Figure 1.1 Permanent Magnet Applications in a Typical Automobile

Ferrite, a magnet material developed in the 1950's, is currently the dominating permanent magnet material used in automotive applications, with a $(BH)_{max}$ of about 2 to 4 MGOe. There are two new developments that enable the application of much higher

performance ((BH)_{max} of 8 to 12 MGOe) permanent magnetic material in the transportation industry. One is the development of new nano-structured Neodymium-Ferrite-Boron (Nd-Fe-B) intermetallic permanent magnetic materials. Another is the use of the injection molding process to mass-produce bonded Nd-Fe-B permanent magnets, making them cost competitive with the traditional Ferrite. The use of such high (BH)_{max} permanent magnets could increase the efficiency and reduce the weight of motors and actuators on cars and trucks. This, in turn, can improve the vehicle's fuel efficiency. These injection molded Nd-Fe-B magnets could also have widespread impact upon the energy savings and performance of electrical motors used in various other industries.

1.1 MAGNETIC MATERIAL DEVELOPMENT

High-energy magnets based on $Nd_2Fe_{14}B$ were first reported in late 1983 [1,2]. Since then, much research has been devoted to the improvement of the intrinsic material properties and the development of fabrication techniques to use various rapid solidification methods to produce the desired nano-structured Nd-Fe-B powders.

The Nd₂Fe₁₄B intermetallic compound is the main phase of the magnetic material. Alloys with nominal compositions near the stoichiometric Nd₂Fe₁₄B are prepared by melt spinning or jet casting, as illustrated in Figure 1.2, to achieve a 10^6 °C/sec or faster cooling rate. This rapid cooling reduces the α -Fe phase, maximizes the Nd-Fe-B phase, and allows the desired magnetic properties.



Figure 1.2 Jet Casting

The ribbons or flakes that are produced are then ground to particles of less than 40 ANSI mesh size. Afterwards, a controlled thermal treatment is usually applied to develop appropriate microstructure and desired magnetic properties. Because of the fine crystalline size, powders obtained are typically magnetically isotropic. This isotropy can be converted into anisotropic characteristics by using a hot plastic deformation process to press the NdFeB powder at an elevated temperature above 700 °C as shown in Figure 1.3.



Figure 1.3 Hot Plastic Deformation Process

Anisotropic powder should yield magnetic properties of much higher $(BH)_{max}$, if an appropriate magnetic field is used to align the crystals to a preferred direction. (For the Nd₂Fe₁₄B type materials, the c-axis is the preferred direction.) Because most of the crystals are aligned in the same direction, a significantly higher B_t and, consequently, $(BH)_{max}$ can be obtained when compared to isotropic powder. To gain the full advantage

of this anisotropic characteristic, powders also need to be aligned to their preferred orientation by applying a magnetic field of sufficient strength (> 10 kOe) during the injection molding process. Thus, the aligning magnetic field configuration and the mold design are important.

The Nd-Fe-B type magnets can be typically classified in two categories: metallic and bonded. Figure 1.4 shows examples of metallic and bonded permanent magnets. The metallic magnet can be produced either by the conventional alloy casting and powder metallurgy method or by a technique involving hot pressing and die upsetting. In general, metallic Nd-Fe-B magnets are fully dense and anisotropic in nature with (BH)_{max} ranging from 25 to 45 MGOe. Metallic Nd-Fe-B magnets do, however, require grinding steps to bring them into the required shape and dimensional tolerances. A surface coating is also required to prevent environmental corrosion or degradation. These two steps usually contribute a significant portion to the magnet production costs. Further, the finishing grinding steps limit their usage in applications where complicated geometry is required.



Figure 1.4 a Example of Metallic Permanent Magnets



Figure 1.4 b Example of Bonded Permanent Magnets

1.2 INJECTION MOLDING DEVELOPMENT

Injection molding offers an efficient means to produce magnets of near net shape and low cost. This process offers great potential for cost reduction for the electromotors and actuators industry. Net shape manufacturing offers the advantage of high-volume production of magnets with accurate dimension tolerances. Moreover, polymer binders, if properly selected, can also serve as an insulator to isolate the magnetic powder from exposure to the environment and act, therefore, as a protection against possible corrosion.

Unlike metallic magnets, bonded magnets require a binder to "glue" magnetic powders together. The Nd-Fe-B magnetic powder is mixed with the thermo-set or thermo-plastic type polymer/resin and other additives. This mixed powder is then kneaded and pelletized before injecting to a mold.

The reciprocal screw rotating in a heated barrel, as illustrated in Figure 1.5, is used to inject the molten mixture of magnetic powder and polymer resin to a mold.



Figure 1.5 The Reciprocal Screw Process

As shown in Figure 1.6, the mold has runner, secondary runner, spruce, and gate to direct the material to the cavity, which is shaped to match the part geometry. Multiple parts, all with near net-shape geometry, can be made in each injection stroke. This has made the injection molding process ideal for high volume production for the transportation industry.



Figure 1.6 Details of the Injection Mold

There are two means to increase the $(BH)_{max}$ of bonded magnets. One is to increase the volumetric percentage of the magnetic powder in the bonded magnet. The other is to use an anisotropic powder and to utilize a magnetic field to align the magnetic powder during the injection molding process. Increasing the concentration of particles, however, will weaken the strength of bonded magnets.

Two commonly used polymer resins to mix with magnetic powder for injection molding are Polyamide (Nylon) and Polyphenylenesulfide (PPS). PPS has better toughness at elevated temperature (180 °C) than Nylon. However, since PPS has higher viscosity, injection molding of a PPS/NdFeB compound is more difficult. The screw and mold clamping forces are higher, the operating temperatures in the barrel and mold are higher, and wear of screw and mold are more prominent.

1.3 PREVIOUSLY CONDUCTED TESTS

In addition to the magnetic properties, the mechanical properties such as the tensile and bending strength, as well as Young's modulus are also of importance to automotive applications. To assure non-failing operations in automotive applications, the injection molded magnets must meet certain minimum mechanical strength requirements within the entire operation temperature range, typically from -40 to 180 °C.

The magnetic properties of bonded magnets have been previously studied [3,4]. However, the design of magnets for motors and actuators does not just encompass the selection of magnetic properties. Mechanical properties at cryogenic and elevated operating temperatures are just as crucial in the correct choice of material. This data, however, is not available for newly developed Nylon-based injection molded Nd-Fe-B magnets. Previously, mechanical properties of sintered Nd-Fe-B type magnets have been researched. The typical properties determined encompassed the bending strength [5], fracture toughness [6], and uniaxial tensile strength [7] of sintered Nd-Fe-B magnets. Ikuma et al. [8] measured the shear strength of extrusion-molded Nd-Fe-B magnets. Xiao and Otaigbe [9] studied the effect of liquid crystal polymer and surface modification on mechanical properties of PPS bonded magnets.

1.4 MECHANICAL TESTS TO BE CONDUCTED

The objective of this study was to determine the mechanical properties of Nylon bonded Nd-Fe-B specimens according to ASTM standards D638-99 [10] for tensile tests, D790-99 [11] for flexural bending tests, and C1259-98 [12] for dynamic Young's modulus measurements. Two types of Nd-Fe-B particles were studied. One type of particle was of irregular plate shape; the other was of spherical shape. Tensile strength was tested at –40, 23, and 100 °C to investigate the strength of Nylon bonded magnets at all operating temperatures. PPS-based magnets were analyzed at –40, 23, 100, and 180 °C. Only room temperature (23 °C) tests were conducted for the bending and dynamic Young's modulus tests. SEM was used to examine the fracture surface and gain insight into the failure mechanism.

2 TEST SETUP

2.1 ASTM-STANDARDS

2.1.1 Tensile Tests (at various temperatures)

To test the tensile properties of the materials, ASTM Standard D 638-99 [10] was used. This testing method can be applied to unreinforced and reinforced plastics in the form of dumbbell-shaped test specimens under defined conditions of pretreatment, temperature, humidity, and testing machine speed. The specimens were held in place by hydraulic grips. The loadcell had a capacity of 100 kN. The material to be tested was injection molded into the geometry of specimen Type IV as shown in Figure 2.1. This type was chosen since the materials were expected to behave like nonrigid plastics and since the thickness of the specimens did not exceed 4 mm.



Figure 2.1 Type IV Tensile Specimen

2.1.2 Bending Tests

The ASTM Standard D 790-99 [11] was chosen to determine the flexural properties of the Nd-Fe-B compounds. In context with the standard, a four-point bend test was selected. The main difference between the three-point bend and the four-point bend is the location of the maximum bending moment and thus, the maximum axial stresses. In the 3-point bend, the maximum stresses occur on a line under the loading nose, whereas in the 4-point bend the maximum stresses can be found over an area between the loading noses.

2.1.3 Ultrasonic Determination of Young's Modulus

To determine the dynamic Young's modulus, ASTM Standard D 1259-98 [12] was applied. This method makes the determination of dynamic elastic properties possible. It can be applied to advanced isotropic ceramics at ambient temperatures. This test is based on the fact that advanced ceramics possess specific mechanical resonant frequencies in the flexural mode of vibration that are directly related to Young's modulus, mass, and geometry of the test specimen. Now, since mass and geometry are known, and the resonant frequency can be measured with the correct apparatus, the elastic modulus can easily be determined. The specimens used in this test were of the same dimensions as the ones used for the bending tests. Figure 2.2 shows the Grindo-Sonic MK4i tester that was used for the measuring of the frequency, as well as the test setup.



Figure 2.2 Test Set-up for Dynamic Modulus Testing

2.2 INJECTION MOLDED MATERIALS TO BE TESTED

Injection molding of Nd-Fe-B type magnets offers an economically efficient means to produce magnets of any shape. Polymer binders, such as Polyamide-11 (Nylon-11) or Polyphenylenesulfide (PPS) can serve as a protection against possible corrosion and, more importantly, will increase the mechanical properties of the material. The steps necessary to form magnets of various shapes through the injection molding process are shown in Figure 2.3. First, the NdFeB powder is mixed with the synthetic resin (i.e. Nylon or PPS) and other additives. This compound is then kneaded and pelletized before it is being injected into a mold. During the molding, a magnetic field is applied to generate anisotropic magnets. This anisotropy, however, is only applicable to the magnetic properties, not to the mechanical properties of the magnetized.



Figure 2.3 Flow Chart of the Injection Molding Process

Eight of the tested materials contained irregular plate shape Nd₂Fe₁₄B particles (150 μ m x 150 μ m x 30 μ m) embedded in either a Nylon or a PPS matrix. Another magnetic material tested contained spherical Nd₂Fe₁₄B particles with a diameter of 30 μ m, embedded in a Nylon matrix. The irregular plate shape Nd-Fe-B particles (Figure 2.4 (a)) were produced through the melt-spinning and grinding methods. This procedure creates thin plates of about 35 μ m thickness. The size of the flakes varies significantly from as small as 10 μ m to as large as 500 μ m. The spherical particles (Figure 2.4 (b)) were produced with the atomization method. Their sizes vary as well, ranging from 5 μ m to 50 μ m diameters. These two types of particles were injection molded at different volume percentages to create tensile and flexural specimens.



Figure 2.4 SEM Micrographs of the Two Types of Nd-Fe-B Particles Used in this Study (a) Irregular, Plate Shape Particle Made by Melt Spinning and Grinding and (b) Spherical Shape Particle Made by Atomization.

The chosen numbering scheme reflects the volume percentage of particles to matrix. For example, PPS517 is a PPS matrix with 51.7 volume % of $Nd_2Fe_{14}B$ particles. The volume percent was calculated using specific densities of particles and matrix. Table 2.1 below summarizes the tested materials.

Numbering Scheme	Matrix	Estimated vol-%
Ny597	Nylon-11	59.7
Ny599	Nylon-11	59.9
Ny604	Nylon-11	60.4
Ny710	Nylon-11	71.0
NyZK620 *	Nylon-11	62.0
PPS503	Polyphenylenesulfide (PPS)	50.3
PPS517	Polyphenylenesulfide (PPS)	51.7
PPS610	Polyphenylenesulfide (PPS)	61.0
PPS614	Polyphenylenesulfide (PPS)	61.4

Table 2.1Numbering Scheme

* denotes spherical shape particles, all others contain irregular plate shape particles

2.3 GEOMETRY OF SPECIMEN

The specimens for the tensile and bending tests were injection molded based on the specified dimensions in the two ASTM standards (D 638-99 and D 790-99) [10,11]. Figures 2.5 and 2.6 below show dimensioned drawings of the tensile and the bending specimen, respectively.



Figure 2.5 Dimensioned Drawing of Tensile Specimen (Type IV)

Figure 2.6 Dimensioned Drawing of Flexure Specimen

2.4 DIFFERENT TEMPERATURE TESTS

The higher and lower temperature tensile tests were carried out based on ASTM Standard D 638-99 [11] as well. An Applied Test Systems (ATS) furnace was used in combination with an ATS PID-controller (see Figures 2.7 and 2.8.) Since the materials were expected to yield under much lower load, the loadcell for this test format was chosen with a capacity of 1000 N.



Figure 2.7 Set-up for Tensile Tests at Elevated Temperatures



Figure 2.8 Interior of Furnace

2.4.1 Manual Grips

The ATS furnace made the use of manual grips necessary. These grips were machined from steel in two parts. The base was secured to threaded rods, which in turn were connected to the base of the MTS tester and the cross-head. The base as well as the cover were serrated to assure better grip of the specimen. The cover was held in place by two screws. Lock-washers and anti-seize compound were used to assure smooth operation under the varying load conditions. Figure 2.9 shows a pure Nylon specimen clamped into the manual grips before mounting it onto the MTS tester. Figures 2.10 and 2.11 show the connection from the threaded rod to the cross-head and the mounted specimen in the interior of the furnace.



Figure 2.9 Pure Nylon Specimen held by Manual Grips



Figure 2.10 Connection of Threaded Rod to Crosshead



Figure 2.11 Utilization of Manual Grips

2.4.2 Elevated Temperature Tests

In order to determine the time needed for the specimen to reach the necessary temperatures, one thermocouple was mounted onto the gauge section of the specimen and one thermocouple was placed into the furnace to monitor temperature increase. The high fluctuations in temperature through the overshoot of the PID controller made a higher setpoint of 113 °C necessary for the 100 °C tests. Time and temperature were monitored to find the delay until the furnace and the surface of the specimen had reached a stable 100 °C. This point was reached after 15 minutes. An additional 5 minutes were granted for the specimen to uniformly reach 100 °C. Thus, 20 minutes were set aside before each material could be tested.

The same procedure was followed to determine the time-delay for the 180 °C tests. Here, the setpoint of the controller was chosen to 195 °C. Again, 20 minutes were deemed sufficient for the specimen to reach a uniform temperature distribution of 180 °C.

2.4.3 Cryogenic Temperature Tests

The same Applied Test Systems (ATS) furnace and ATS PID-controller were used for the low temperature tests (-40 °C) of the compounds. The furnace was connected to a liquid Nitrogen (LN) bottle through a thermally insulated rubber hose. Since the overshoot of the controller could now be restricted through the manually adjusted valve on the LN tank, the setpoint on the controller was selected to be -42 °C. Again, the time was monitored until the specimen reached a uniform temperature. This was achieved after 10 minutes. Figures 2.12 and 2.13 below show the setup for the cryogenic testing. The black hose in the picture is the connection to the liquid nitrogen tank. Figure 2.12 shows the ATS controller in the background.



Figure 2.12 Set-up for Tensile Test at Cryogenic Temperature



Figure 2.13 ATS Controller and Liquid Nitrogen Tank

2.5 **BENDING TESTS**

The four-point flexural tests were performed on a MTS 120 testing machine. To control the crosshead speed and to measure the deflection of the specimen under load, an INSTRON 8500 controller was used. The crosshead speed was set to 0.01 mm/s. Figure 2.14 shows the fixture for the 4-point bend test.



Figure 2.14 Fixture for Flexure Tests

2.6 ULTRASONIC TESTS

The same specimens that were used for the bending tests were utilized in the ultrasonic determination of the dynamic modulus. The specimens were placed onto two rubber supports, spaced apart as described in the applicable ASTM standard [12]. Figure 2.15 shows a dimensioned drawing of the correct setup. This simply supported beam was then tapped with a small metal hammer and probed to measure the frequency response.

Since the same specimens that were used for the bending tests were utilized in the ultrasonic determination of the dynamic modulus, it was impossible to test for the shear modulus as well, and thus for Poisson's ratio. Poisson's ratio, however, is necessary to analyze the data. Therefore, different values for Poisson's ratio in a range from 0.2 (ceramics) [13] to 0.4 (Nylon and/or PPS) [14] were selected and Young's modulus was determined for each of those values.



Figure 2.15 Dimensioned Drawing for Ultrasonic Setup

3 ANSYS ANALYSIS

While the MTS extensometer delivered very repeatable and reliable data from the tests carried out at room temperature, its response taken at elevated and cryogenic temperatures proved not dependable. During testing, it was already realized that at elevated temperatures the extensometer slipped numerous times. The way of affixing the extensometer to the test specimen was the reason for the slipping. The extensometer was attached to the sample via rubber bands, which was sufficient at room temperature. At elevated temperatures, however, rubber stretches and the extensometer slipped therefore. A clip-on extensometer would have given more reliable data, as the preliminary testing showed. The clip-on extensometer, however, was not for the final testing.

At cryogenic temperatures, the extensometer froze up. This could have been avoided by using a furnace with humidity control. Unfortunately, such a furnace was not accessible for testing.

The issues with the extensometer left the strength data to be presented relative to the displacement of the cross-head. Such data, however, is customarily presented in relation to the percent strain the specimen undergoes. Since the cross-head displacement data was reliable, the gauge-length of the specimen had to be found.

It was thus decided to develop a finite-element model of the sample, apply a prescribed displacement at the end-nodes (where the grip would be holding the specimen) and calculate the stress in the specimen. Applying Hooke's Law and the Stress-Strain relationship, the gauge-length of the samples was determined.

The finite-element analysis provided not only means to calculate the gauge-length, but showed stress-concentrations in the specimen. This stress-concentration was noticeable during testing, as all specimens failed in the transition between gauge-area and grip-area. Additional finite-element models were constructed to counteract the effect of stress-concentration through a change in geometry of the test specimen.

3.1 CONSTRUCTION OF THE MODEL

To simplify the analysis, a quarter-model of the specimen was constructed. To do this, the correct geometry was laid out in AutoCad. The key-points were taken from that model, and a quarter of it was modeled in ANSYS. Figure 3.1 below shows the quarter model as input into ANSYS.



Figure 3.1 Quarter Model of the Type IV Tensile Specimen for ANSYS

Edge AB of this model was constrained with rollers in the x-direction and fixed in the y-direction. The right hand side (BD) was modeled with rollers in the y-direction and fixed in the x-direction. The section of the left side (AC) that would be held by the grip (of a length of 17 mm) was replaced with a prescribed displacement of 0.1 mm in the negative x-direction (to the left).

Plane2 triangular structural solid elements were chosen in ANSYS over Plane42 structural solid elements. Both elements can be used for 2-D modeling of solid structures by invoking either the plane stress or the plane strain option. The nodes that make up each element have two degrees of freedom each: one in the x-direction and one in the y-direction. The displacement behavior of the Plane2 elements is quadratic, much like the Plane42 elements. Plane2 elements, however, are 6-node triangular with 12 degrees of freedom and thus are better suited for irregular meshes than the Plane42 elements, which are 4-node rectangular.

The model was generated with a mesh of 442 elements. The ANSYS built in h solver analyzed the problem as a static, structural analysis with plane stress option.

3.2 CALCULATING THE GAUGE LENGTH

Since the materials were expected to have different moduli of elasticity, a range for Young's modulus was chosen. The Poisson's ratio of the tested compounds was unknown as well, so there too, a range was selected. The ANSYS model was run several times with fixed geometry, fixed constraints, and fixed applied displacement. The only changing variables were Young's modulus and Poisson's ratio. The stresses resulting from these analyses were recorded and tabulated as can be seen in Table 3.1 below.

Stress (MPa)	Poisson's Ratio		
Young's Modulus (GPa)	0.2	0.3	0.4
0.7	2.3303	2.3303	2.3303
1.8	5.9921	5.9921	5.9921
2.9	9.6539	9.6539	9.6539
4.0	13.3160	13.3160	13.3160

Table 3.1Maximum Stresses from ANSYSAnalysis of Type IV Test Specimen

Since it was known that the material behaved elastically at very small applied displacements, Hooke's law and the definition of strain could be applied to find the gauge length of the specimen.

From Hooke's law, we know that:

$$\boldsymbol{s} = \boldsymbol{E}\boldsymbol{e} \tag{1}$$

where s = stress in MPa; E = Young's Modulus; and e = strain.

By definition, the strain is given by:

$$\boldsymbol{e} = \frac{\Delta l}{l} \tag{2}$$

where $\varepsilon = \text{strain}$; $\mathcal{P}l = \text{change in length}$; and l = original gauge length of the specimen.

Substituting the definition for strain into Hooke's law and solving for the original gauge length l of the specimen, yields the following expression:

$$l = E \frac{\Delta l}{s} \tag{3}$$

Now, the change in length was the applied displacement, d. However, since only a quarter of the actual specimen was modeled, this displacement had to be doubled before it could be substituted in to the above equation. Therefore:

$$\Delta l = 2d \tag{4}$$

where d = applied displacement in the x-direction.

Thus, the gauge length of the specimen could now be determined through the following expression:

$$l = E \frac{2d}{s} \tag{5}$$

Table 3.2 below gives a summary of the calculated gauge length for varying Young's modulus and Poisson's ratio.

Gaugelength (mm)	Poisson's Ratio		
Young's Modulus (GPa)	0.2	0.3	0.4
0.7	60.0781	60.0781	60.0781
1.8	60.0791	60.0791	60.0791
2.9	60.0793	60.0793	60.0793
4.0	60.0781	60.0781	60.0781

 Table 3.2
 Summary of Calculated Gaugelength in mm

The values in the above table were averaged and the standard deviation was calculated. The gauge length of the tensile specimen was thus determined to be 60.0787 mm, with a standard deviation of 0.0005936 mm.

3.3 STRESS CONCENTRATION IN THE ASTM MODEL

3.3.1 Current Type IV Test Specimen

The ANSYS analysis of the Type IV tensile test specimen not only provided the necessary data for the strain calculations, but also supported a failure phenomenon which became apparent during testing. All of the tensile specimen, whether Nylon or PPS based, fractured during cryogenic and room temperature tests in the same area, namely, in the area of stress concentration. This stress concentration arises through a change in geometry from the slender test section to the larger section at which the grips are attached. Figure 3.2 below shows a fractured sample.



Figure 3.2 Fractured Ny597 Specimen

Figure 3.3 below shows the von-Mises-Stress distribution in a quarter model of the Type IV tensile specimen. The stress concentration in light blue is clearly visible.



Figure 3.3 Von-Mises Stress Distribution in the Type IV Quarter Model



Figure 3.4 Von-Mises Stress Distribution in the Type IV Half Model
To verify the validity of the quarter model, as well as to check the magnitude of the stress concentration, one half of the specimen was modeled. Figure 3.4 above shows the result of this analysis with the same prescribed displacement, the same Young's Modulus and the same Poisson's ratio as chosen for the quarter model of Figure 3.3 above.

Figure 3.5 below shows a close-up of the area of stress concentration. The stress concentration factor (K_f) that resulted from this analysis lies at 1.08 and was calculated by comparing the nominal stresses in the gauge-section to the maximum stress at the point of stress concentration as shown in Figure 3.3.



Figure 3.5 Close-up View of the Area of Maximum Stress in the Type IV specimen

3.3.2 Stress Concentrations on all ASTM D 638-99 Specimens

In addition to Type IV specimens, ASTM standard D 638-99 offers three other shapes. Type I and II are specimens to be used for materials with a thickness of less than 7 mm, whereas Type III specimen are required for materials with a thickness between 7 and 14 mm. Type I specimen must be used to test reinforced composites and are the preferred form for rigid and semirigid plastics. Type II samples may be used if Type I samples do not break in the narrow section. Type III specimens must be used for all materials with a thickness greater than 7 mm. As mentioned above, Type IV specimens are only permitted for testing nonrigid plastics with a thickness of 4 mm or less.

The finite-element analysis of the different specimens was carried out in the same manner as for the Type IV sample. The same Plane2 elements were chosen with the plane stress option. The models were generated with a mesh of 442 elements. The ANSYS built in h-solver analyzed the problem as a static, structural analysis. The same Young's modulus, Poisson's ratio, and thickness were used, and the same boundary conditions were applied.

Table 3.3 below shows the different contour plots reflecting the von-Mises stresses of the various test specimens and summarizes the stress-concentration factors. As was to be expected, Type II specimens show the smallest stress-concentration factor due to the large radius in the transitional area.

ASTM 638-99 Samples	Contour-Plots (Von-Mises Stress in MPa)	Stress- Concentration Factor K _f
Type I	900 - 26001 900 - 26334 36101 36191 203912 36372 26332 36776 26376 26376 26376 26376 26376 26376 26376	1.046
Type II	5000 305 -0600 305 -06210 5093 5093 5093 10670 10670 10776 120776 121578 25676	1.014
Type III	9401 ~41471 9407 ~219904 9407 ~219904 9404 94040 94040 94040 94040 140405 140501 140405 140501 140405 140501 140405 140501 140500 140500 140500 140500 140500 140500 140500 1405000 14050000000000	1.068
Type IV	1965 * _101822 1965 *_101822 1967 *=05495 1977 *=05495	1.119

Table 3.3Contour-Plots and Stress Concentration Factors of ASTMD 638-99 Test Specimens

3.3.3 Re-Design of Type IV Test Samples

Following the good results obtained in the analysis of Types I through III, the next step was to re-design the current Type IV sample. The simpler geometry with one radius only of Types I through III was especially appealing, since it would simplify manufacturing of the specimens drastically. Therefore, the first modification involved a change to a simpler geometry with only one radius. Overall length, overall width, narrow section length and width were left as described by ASTM standard D 638-99. Thickness, Young's modulus, Poisson's ratio, and prescribed displacement were held constant, as was the mesh of the specimen.

3.3.3.1 Single Radius Modifications and Stress-Concentration Factors

Four different models (Figure 3.6 below) were analyzed in ANSYS with radii ranging from 6.35 mm to 39.627 mm. Table 3.4 below summarizes the analysis results of the stress-concentration factors, as well as the contour-plots of the different models. As can be seen in the table below, an increase in radius larger than 28.5 mm contributes only minimally to the reduction of the stress-concentration factor K_f . From this it can be concluded that for this kind of geometry, the smallest resulting stress-concentration factor would be 1.02.



Figure 3.6 Quarter Model with One Radius

	Radius (mm)	Contour-Plots (Von-Mises Stress in MPa)	Stress- Concentration Factor K _f
SI	6.350	8MN #3120 8MN #3120 800472 3120 67292 259410 325942 329415 329415 32942 34045 32942 34942 3494	1.267
SII	17.450	SHH -1141 SHK -1	1.096
SIII	28.500	SNN +9634 ENX +437280 9634 57150 144666 152183 139699 247215 294731 342246 343764 437280	1.058
SIV	39.627	10821 20821 20821 20827 20807	1.042

Table 3.4Contour-Plots and Stress Concentration Factors of
Modified Test Specimen with One Radius

3.3.3.2 Double Radius Modifications and Stress-Concentration Factors

The second modification was based on the original Type IV specimen which utilized two radii in the transition from the narrow gauge section to the larger grip section of the samples. Five different models were analyzed in ANSYS. Figure 3.7 below shows a close-up drawing of the transition area, clearly identifying the two radii.



Figure 3.7 Quarter Model with Two Different Radii

From rules of geometry, the two angles that describe the arclength have to be equal and can thus be related through the following equations:

$$(RI)\cos q = RI - (6.35 - y)$$
(6)

$$(RO)\cos q = RO - y$$
(7)

$$(RI)\sin q = 21.515 - x$$
(7)

$$(RO)\sin q = x$$

where: RI = inner radius, RO = outer Radius, y = vertical coordinate of the point of intersection of arcs, x = horizontal coordinate of the point of intersection of arcs

Setting the equations from set (1) equal to each other and solving for *RO* yields:

$$RO = \frac{yRI}{6.35 - y} \tag{8}$$

Doing the same to equation set (2) and solving for RI yields:

$$RI = \frac{21.516 - x}{x} RO \tag{9}$$

Substituting (4) into (3) and solving for x yields:

$$x = \frac{21.516}{6.35} y \tag{10}$$

Using Pythagoream theorem and substituting equation (5) for x yields:

$$RO^{2} = (RO - y)^{2} + x^{2}$$
(11)
$$y = \frac{2(6.35)^{2}}{21.516^{2} + 6.35^{2}} RO$$

Substituting (6) back into (5), and the resulting expression back into (4), yields the following relationship between the two radii:

$$RI = \frac{21.516^2 + 6.35^2}{12.7} - RO$$
(12)

Table 3.5 below summarizes the selected radii for the modification, the corresponding contour-plots, and the corresponding stress-concentration factors. Model D1 is the current Type IV specimen. As was expected, the model with the larger radii at the transition point from narrow gauge-section to larger grip-section shows the lowest stress-concentration factor.

	Radius	Radius		Stress-
	RI	RO	Contour-Plots (Von-Mises Stress in MPa)	Concentration
	(mm)	(mm)		Factor K _f
DI	14.224	25.400	2NN =108728 8902 =485985 10872 = 485985 10872 = 485985 10872 = 485985 10872 = 485985 10872 = 485985 10872 = 485985	1.119
DII	16.627	23.000	INN -103809 INX -473931 103809 144934 166059 227183 266306 399433 350557 391682 433867 473931	1.113
DIII	19.816	19.816	SEM =96091 SEX =96091 SEX =461629 96391 126973 177555 218137 29301 339883 380465 421047 461629	1.084
DIV	22.627	17.000	SNN -88810 8MX -852639 00010 129213 169517 210020 250423 290826 331229 371633 471633 471633	1.073
DV	25.400	14.224	SHM =82981 SHM =82981 SHX =468908 82981 125862 168743 211623 254504 297385 340265 340265 383147 425028 468908	1.090

Table 3.5Contour-Plots and Stress Concentration Factors of
Modified Test Specimen with Two Radii

4 RESULTS AND DISCUSSION

4.1 AVERAGING

Five tensile tests were carried out for each compound of Table 2.1 at different temperatures. In addition, some of the compounds were tested in bending. There, seven tests were performed. In order to find representable data, the results for each compound were compared. Test results that fell outside a range of acceptable data were discarded. Figure 4.1 shows the averaging process of PPS614 at room temperature. The data was averaged up to the point were the first specimen broke. This resulted in presenting the lowest possible ultimate tensile strength. The data resulting from this averaging procedure enabled a comparison of the different compounds at various temperatures as well as a comparison of the varying strength with changing temperature per compound.

The bending results were averaged in the same manner to yield a comparison of bending strength in different compounds with regard to their volume percentage of particles.



Figure 4.1 Averaging Procedure shown on PPS614

4.2 TENSILE RESULTS

Four different temperature tests were carried out for each PPS compound of Table 2.1. Unlike PPS based compounds, Nylon based compounds could only withstand temperatures up to 100 0 C. Nylon based injection molded magnetic materials containing irregular particles were tested at only three different temperatures (-40 0 C, 23 0 C, and 100 0 C), while the compound containing spherical particles was tested at four temperatures.

The values represent the arithmetic average of all the measurements made for each compound. The sample standard deviation for the strength at any strain did not exceed 5.63 MPa, which indicates that the samples were of uniform quality and the tests were reproducible. The presented values are engineering stresses and strains.

4.2.1 Tensile Results for Nylon-Compounds

Figures 4.3 through 4.8 reflect a comparison of tensile strength vs. strain at different testing temperatures for each Nylon-based compound. Figures 4.9 through 4.12 show the relationships of tensile strength and strain for the different compounds at each esting temperature.

More than 60% reduction on the tensile strength at high temperatures for Nylon based compounds was observed. For higher temperatures (80 °C and 100 °C), the amorphous regions of the nylon matrix deform as a thermoplastic, while the crystalline regions deform as a rigid solid [15]. The apparent strain hardening effect is due to the increased flow resistance of the matrix as the polymer chains elongate and transfer load to the crystalline regions. There is also a severe constraint due to the high volume fraction of the Nd-Fe-B particles. Localized strains in ligaments surrounding the particles can be significantly larger than the nominal strain.

The tensile strength for Ny597 and NyZK620 at -40 °C is increased by over 50% relative to that of room temperature. This can be explained with the transition of the Nylon bond to a more brittle, glassy phase at low temperature. At cryogenic temperature, the difference in thermal expansion coefficient of Nylon (2.3 -11x10⁻⁵ /°C) [16] and Nd-Fe-B (4 - 8x10⁻⁶ /°C) [17] generates a compressive residual stress on the Nd-Fe-B particle by the surrounding Nylon bond. This compressive residual stress on the interface between the matrix and Nd-Fe-B particle can help improve the tensile strength of Nylon injection molded magnets at cryogenic temperature.

The ultimate tensile strength increases in general with increasing volume percentage of Nd-Fe-B particles. The compounds with higher percentage of Nd-Fe-B, Ny710, showed overall much lower strength compared to that the other two compounds. One compound with 59.9 vol% of Nd-Fe-B particles behaved unpredictably. To follow the general trend, it was expected that its ultimate tensile strength would fall within that of Ny604 and that of Ny597. Instead it exceeded the tensile results of Ny604. Scanning Electron Microscopy showed that Silicon-Carbide-Fibers were used as a reinforcement of the matrix in this particular compound.

In comparison to the Nylon-11 based compounds containing rectangular particles, a Nylon-11 matrix surrounding spherical Nd-Fe-B particles with a concentration of 62 vol-% was tested. This compound was tested at four temperatures (-40 0 C, 23 0 C, 80 0 C and 100 0 C). As can be seen in Figure 4.6, this combination behaves in a much more ductile manner than the other tested compounds. The overall tensile strength, however, has been

drastically reduced at all tested temperatures. The difference in the behavior of the compound at -40 and 23 0 C as compared to the behavior at 80 and 100 0 C can be explained with the glass transition temperature of Nylon-11. This temperature lies at around 50 0 C [16] and is the reason for the shift from brittle to ductile behavior. A strain hardening effect comes into play at elevated temperatures as well and explains the increased tensile strength at 80 and 100 0 C. The difference in the thermal expansion coefficient of Nylon-11 [16] and Nd-Fe-B [17] particles explains why the material exhibits higher tensile strength at cryogenic temperature in comparison to the results at room temperature.

Pure Nylon-11 was tested as well. This offered a verification of the tensile results, as the ultimate tensile strength of this material is openly available. This material was tested at four temperatures (-40 °C, 23 °C, 70 °C and 120 °C). Figure 4.8 shows a comparison of the test results at these temperatures. At room-temperature the specimen did not reach the maximum tensile strength. Rather, the tests were interrupted because the maximum range of the tensile tester was reached. The data gathered from the cryogenic temperature test resulted in an expected curve. From the curves of the elevated temperature test data it can be clearly seen that a material hardening effect as a function of strain set in after about 10 % strain were achieved. The failure mode at these elevated temperatures is different as well. A relative linear increase in strength with increased strain points toward necking of the material, which was clearly visible during the test as well. At all temperatures, the strain to fracture for the NyZK620, the compound containing spherical particles (Figure 4.7) is much larger than for Nylon-based compounds containing irregular shape particles (Figures 4.3 through 4.6). This is illustrated in pictures of the deformed test samples in Figure 4.2. Different levels of strain can be seen on Ny597, NyZK620, and Ny tensile specimen tested at 100 °C. The Ny597 sample shown in Figure 4.2(a) has small strain to fracture. This specimen still resembles the original geometry of tensile test specimen. Figure 4.2(b) illustrates the relatively large deformation (15% strain) achieved on the NyZK620 with spherical particles. As shown in Figure 4.2(c), the pure Nylon, Ny, sample continued to deform and did not fracture at the end of test when 160% strain was attained.



Figure 4.2 Pictures of three fractured or deformed tensile test specimens tested at 100 °C (a) Ny597, (b) NyZK620, and (c) Ny

The very large effect of particle shape on the strain-to-fracture at about the same volume fraction of particles (NyZK620 vs. NY597) is surprising. The sharp angular particles, and perhaps also the fine dispersion of very small angular particles, evidently initiates very rapid fracture of the matrix ligaments as strain is increased. The particle angularity causes much larger stress intensification compared to the spherical particles. In addition, the higher strength levels reached for the compound with irregular shape particles at the given strain seem to accelerate failure.



Figure 4.3 Comparison of strength at different temperatures for Nylon based compound with 59.7 vol% of irregular Nd-Fe-B particles



Figure 4.4 Comparison of strength at different temperatures for Nylon based compound with 59.9 vol% of irregular Nd-Fe-B particles



Figure 4.5 Comparison of strength at different temperatures for Nylon based compound with 60.4 vol% of irregular Nd-Fe-B particles



Figure 4.6 Comparison of strength at different temperatures for Nylon based compound with 71.0 vol% of irregular Nd-Fe-B particles



Figure 4.7 Comparison of strength at different temperatures for Nylon based compound with 62.0 vol% of spherical Nd-Fe-B particles



Figure 4.8 Comparison of strength at different temperatures for the matrix (pure Nylon 11)



Figure 4.9 Comparison of strength at -40 ⁰C of Nylon based compounds with different concentration of irregular and spherical Nd-Fe-B particles



Figure 4.10 Comparison of strength at 23 ⁰C of Nylon based compounds with different concentration of irregular and spherical Nd-Fe-B particles



Figure 4.11 Strength at 80 ⁰C of Nylon based compounds with spherical Nd-Fe-B particles



Figure 4.12 Comparison of strength at 100 ⁰C of Nylon based compounds with different concentration of irregular and spherical Nd-Fe-B particles

4.2.2 Tensile Results for PPS-Compounds

The test results confirm that the PPS injection molded magnets can withstand the 180 °C operation temperature required for some automotive applications. However, more than 60% reduction on the tensile strength at high temperatures (100 and 180 °C) for all four PPS compounds is observed.

The tensile strength for PPS503 and PPS517 at -40 °C is increased by about 20% relative to that of room temperature. This can be explained by the transition of the PPS bond to a more brittle, glassy phase at low temperature. At cryogenic temperature, the difference in thermal expansion coefficient of PPS (5.3×10^{-5} /°C) [18] and Nd-Fe-B (4 – 8×10^{-6} /°C) [17] generates a compressive residual stress on the Nd-Fe-B particle by the surrounding PPS bond. This compressive residual stress on the interface between the PPS bond and Nd-Fe-B particle can help improve the tensile strength of PPS injection molded magnets at cryogenic temperature.

The ultimate tensile strength decreases in general with increasing volume percentage of Nd-Fe-B particles. The two compounds with higher percentage of Nd-Fe-B, PPS610 and PPS614, showed overall much higher strength compared to that the other two compounds.



Figure 4.13 Comparison of strength at different temperatures for PPS based compound with 50.3 vol% of irregular Nd-Fe-B particles



Figure 4.14 Comparison of strength at different temperatures for PPS based compound with 51.7 vol% of irregular Nd-Fe-B particles



Figure 4.15 Comparison of strength at different temperatures for PPS based compound with 61.0 vol% of irregular Nd-Fe-B particles



Figure 4.16 Comparison of strength at different temperatures for PPS based compound with 61.4 vol% of irregular Nd-Fe-B particles



Figure 4.17 Comparison of strength at -40 ⁰C of PPS based compounds with different concentration of irregular Nd-Fe-B particles



Figure 4.18 Comparison of strength at 23 ⁰C of PPS based compounds with different concentration of irregular Nd-Fe-B particles



Figure 4.19 Comparison of strength at 100 ⁰C of PPS based compounds with different concentration of irregular Nd-Fe-B particles



Figure 4.20 Comparison of strength at 180 ⁰C of PPS based compounds with different concentration of irregular Nd-Fe-B particles

4.3 BENDING RESULTS

Since the tensile tests had proven that the properties are very similar for similar volume percentage of particles in the matrix, the tests were carried out for only two of the PPS compounds and only three of the Nylon compounds of Table 4.1. Seven bending tests were performed for each material. The averaged results are presented in Figures 4.20 and 4.21. The values represent the arithmetic average of all the measurements made for each compound. The sample standard deviation for the strength at any displacement across the test range did not exceed 5.5 MPa. This indicates that the samples were of uniform quality and the tests were reproducible.

The results of the bending tests follow the general trend that was observed in the tensile tests. The PPS compound with lower volume percent of Nd-Fe-B did withstand not only higher stress but also larger displacement. Compared to the tensile strength data at 23 °C, the bending strength is higher, i.e., 65 vs. 40 MPa for PPS614 and 85 vs. 63 MPa for PPS517.

The bending tests on the Nylon compounds followed the general trend observed from the tensile data as well. The bending strength increased as well with increasing concentration of particles. Compared to the tensile strength at 23 °C, the bending strength is higher here as well, 50 vs. 23 MPa for Ny604, 55 vs. 33 MPa for Ny599, and 40 vs. 20 MPa for Ny597. Due to the non-uniform loading in bending, such trend is commonly observed in comparing the strength results in bending and tensile tests.



Figure 4.21 Comparison of bending strength at 23 ⁰C of Nylon-based compounds with different concentration of irregular shape Nd-Fe-B particles


Figure 4.22 Comparison of bending strength at 23 ⁰C of PPS-based compounds with different concentration of irregular shape Nd-Fe-B particles

4.4 ULTRASONIC RESULTS

Again, due to the similarity in properties, only two of the PPS compounds and only three of the Nylon compounds were considered in this test. Ten frequency readings that fell within a standard deviation of 1.5 were obtained following the ASTM Standard [12] procedure. The Dynamic Young's modulus was then calculated in the following manner:

$$E = 0.9465 \left(m f_f^2 / b \right) \left(L^3 / t^3 \right) T_1$$
(13)

where: E = Young's Modulus; m = mass of bar in g; b = width of bar in mm; L = length of bar in mm; t = thickness of bar in mm; $f_f = fundamental resonant frequency of bar in flexure in Hz; given by:$ $<math>f_f = 2,000,000/R$ (14)

R = Grindo-Sonic reading

 T_1 = correction factor as to be determined according to ASTM Standard [12].

The correction factor T_1 is a function of Poisson's ratio, which was unknown and could not be determined experimentally, since specimen geometry did not allow testing for the shear modulus. Young's modulus was thus calculated using different values for Poisson's ratio, ranging from 0.2 for ceramics [13] to 0.4 for plastics such as Nylon or PPS [14]. Poisson's ratio, however, only contributes minimally to the calculation of Young's modulus. So is, for example, Young's modulus for Ny604 17.63 GPa with a Poisson's ratio of 0.2 and 17.66 GPa with a Poisson's ratio of 0.4. Table 4.2 below summarizes the results of the ultrasonic test, with a chosen value of 0.3 for Poisson's ratio (v) and shows a comparison to the data retrieved from the tensile tests.

Numbering Scheme	Dynamic Modulus	Tensile Modulus	Percent Error
Ny597	15.7 GPa	12.69 GPa	19.20 %
Ny599	35.14 GPa	31.41 GPa	10.62 %
Ny604	17.65 GPa	14.19 GPa	19.58 %
PPS517	24.12 GPa	25.49 GPa	5.67 %
PPS614	31.82 GPa	31.67 GPa	0.46 %

Table 4.1Dynamic Modulus Comparison

The values in the above table represent the arithmetic average of all the measurements made for each compound. The standard deviation of less than 1.5 GPa proves that the tests were reproducible. As stated above, Poisson's ratio only influenced the values for Young's modulus minimally, which is reflected in the percent error of Ny604 that ranges from 19.52 % for v = 0.2 to 19.67 % for v = 0.4.

There is a notable discrepancy in the values of Young's modulus using these two methods. It is likely, that the compound with the softer Nylon exhibits a larger anisotropy than the compound of Nd-Fe-B and PPS and, therefore, does not render itself for accurate measurements of Young's modulus using the dynamic impulse method.

5 SCANNING ELECTRON MICROSCOPY

RCA Laboratory reported the first research model Scanning Electron Microscope (SEM) in 1942 [19]. The first commercial SEM became available in 1964 [20] due to advances in signal processing and amplification. Today, Scanning Electron Microscopy is commonly used to examine fractured surfaces and to determine failure modes.

SEM utilizes a wide, aligned beam of electrons, which scans across an object. Some of the electrons are reflected, others are absorbed by the material as the beam of electrons strikes the object of observation. When electrons are absorbed, the object emits other electrons. Energy and direction of the reflected and emitted electrons are then detected and used to produce a picture of the surface features [20].

For an SEM to work properly, conductive materials must be used, since a build up of net charge is undesirable, causing spots or images that are too bright to be recorded. In order to view surfaces of non-conductive materials, these materials have to be coated with conductive powder (typically gold).

The fractured surfaces were examined using the Scanning Electron Microscope (SEM) to gain insight into the failure mechanism.

5.1 FRACTURED SURFACES OF NYLON-COMPOUNDS

Figure 5.1 show the fracture surfaces of Ny597 in tensile tests at -40, 23, and 100 °C. The irregular plate shape Nd-Fe-B particles can be recognized on the fracture

surface. As described in section 2.2, the plate shape particles are aligned during the injection molding process with the help of a magnetic field. Figure 5.1(c) shows an overall view of the fracture surface. A vortex shape core can be seen in the middle of the sample fracture surface. This vortex is created by the flow and cooling of the matrix and Nd-Fe-B mixture during the injection molding process. The alignment of irregular plate shape particles along the flow direction helps to retain the brittle characteristics of the Nd-Fe-B particles, which is reflected in the results of the tensile and bending tests.

At 23 °C, the Nd-Fe-B particles do not fracture. Instead, the particles are pulled out and leave pockets on the fracture surface, as can be seen in Figure 5.1(d). Figure 5.1(f) shows such particles pulled out or debonded on the fracture surface of a specimen tested at 100 °C. The Nylon is soft at 100 °C, and burrs or extrusions on the fracture surface can be recognized. At -40 °C, the Nylon compound is brittle and the fracture surface is glassy. Pockets due to particle pull-out cannot be easily identified. It is likely that Nd-Fe-B particles fracture at cryogenic conditions.



(d) -40 °C, close-up view

(a) -40 °C



(c) 23 °C

(d) 23 °C, close-up view



(e) 100 °C





Figure 5.2 shows the fracture surfaces of the Nylon compound containing spherical particles (NyZK620) for the tensile tests conducted at -40, 23, and 100 °C. Debonding or separation of spherical particles with Nylon bonds is apparently the failure mechanism. The 62.0 vol-% of spherical Nd-Fe-B particles does not provide structural support, which

makes this type of sample very weak in tensile strength. The transition from a glassy, brittle facture surface at -40 °C to the burr and flash morphology of the fracture surface at 100 °C can also be seen in Figure 5.2.



(b) -40 $^{\circ}$ C, close-up view of C

(a) -40 °C



(d) 23 °C, close-up view

(c) 23 °C



(e) 100 °C

(f) 100 °C, close-up view of D

E Figure 5.2 SEM micrographs of the fractures surfaces of NyZK620 with spherical shape particles

5.2 FRACTURED SURFACES OF PPS-COMPOUNDS

Figures 5.3 (a through h) show the fracture surfaces of PPS503 at four testing temperatures. The vortex shape core that was clearly identified in Figure 4.1 (c) is visible here as well. Again, debonding on the surface between Nd-Fe-B particle and PPS matrix can be identified as the source for failure. Figure 5.3 () shows the pockets that are left in the matrix after the particles are pulled out. There is a notable change in the fractured surface pattern from cryogenic to high temperature tests. At -40 °C, the PPS compound is brittle and the fractured surface is glassy. At 100 and 180 °C, the PPS matrix is softened and PPS extrusions or residual burrs can be observed on the rough fracture surface.



(a) –40 °C

(b) -40 °C, close-up view



(c) 23 °C

(d) 23 °C, close-up view of E



(f) 100 °C, close-up view of F

(e) 100 °C



(g) 180 °C

(h) 180 °C, close-up view of G

Figure 5.3 SEM micrographs of the fractures surfaces of PPS503 with irregular plate shape particles

6 CONCLUSION

Since the development of new fabrication techniques for high-energy magnets based on $Nd_2Fe_{14}B$, much research has been devoted to the improvement of the intrinsic properties of these rare-earth magnets. With the introduction of injection molding as a means to produce near net shape magnets at low production cost, the need has risen for the knowledge of the mechanical properties of these bonded, injection-molded magnets.

The automotive and computer industry typically utilizes these magnetic materials. Therefore, it is essential that the temperature dependent mechanical properties of these compounds be known for a temperature range of -40 to 180 °C. Since these mechanical properties are critical for the design of devices utilizing permanent magnetic materials, tests were conducted to form a database of mechanical characteristics covering the operational temperature range. This will provide valuable information for material designers to tailor the formulation and process parameters to achieve the desired mechanical properties.

Tensile and bending strength, as well as Young's modulus of Nylon-11 and PPS bonded Nd-Fe-B magnets were investigated in this study. Two types of particles were available for testing: irregular plate shape particles with sizes ranging from 10 μ m to 500 μ m, with a thickness of 30 μ m, and spherical particles with diameters ranging from 5 μ m to about 50 μ m. The spherical particles, however, were available for testing only in the Nylon matrix.

The tensile strength for all types of compounds showed strong temperature dependence. Overall, more than 60 % reduction of the tensile strength at high temperatures for either the Nylon-based or the PPS-based compounds was observed. The tensile strength at cryogenic temperatures, however, increased in general by over 50 %.

A strong dependence on the concentration of particles, as well as the type of particles was observed as well. While the tensile strength of PPS compounds decreased in general with increasing volume percentage of Nd-Fe-B particles, it increased for Nylon compounds with increasing concentration. The only exception was Ny599, a fiber enforced Nylon-11 compound with 59.9 vol% of Nd-Fe-B particles. The ultimate tensile strength of NyZK620, the compound with spherical particles, was drastically reduced at all testing temperatures. This compound, however, can easily be identified as an extremely ductile material and could be used for magnetized extruded plastic sheet or other applications were flexibility is highly desirable.

Scanning Electron Microscopy (SEM) analyses indicated that the debonding on the Nd-Fe-B particle and Nylon interface was the main cause of failure at room and elevated temperatures. Fracture of Nd-Fe-B particles was observed on compounds with irregular shape particle at cryogenic temperature.

In this study it was proven that bending tests give reliable enough results to guide in the development of new compounds, as trends that follow the bending tests can be observed during the tensile tests as well. Limitations, however, were observed in using the impulse vibration method to measure Young's modulus of bonded magnets.

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	35x	150x	350x	1000x
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Ny604 23 °C (30x, 130x, 300x, 1000x)				
Ny604 100 °C				
Ny710 –40 °C				

	35x	150x	350x	1000x
Ny710 23 °C (100x, 200x, 700x, 700x)				
Ny710 100 °C				
Ny710 100 °C (failed 5 Mpa under ave.) (35x, 450x, 800x) (800x is a Carbon particle)	54/824 5/84 12 frem 6/5 5/246 /11(2001	50005 5.0V 12 trem #50 5E(A) 711/0001	5X-8X25 5XV 12. Tren XX0 5E[4; 2112227 50.04	
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