

Mechanical properties of polyphenylene-sulfide (PPS) bonded Nd–Fe–B permanent magnets

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Abstract

Mechanical properties, namely tensile and flexural strengths and elastic modulus, of polyphenylene-sulfide (PPS) bonded Nd–Fe–B magnets have been studied from -40 to 180 °C. The ultimate tensile strength (S_{ut}) of PPS bonded magnets decreases with increasing temperature. The tensile strength of PPS bonded Nd–Fe–B magnets was reduced significantly above 100 °C. At 180 °C, the PPS bonded magnets still exhibit a S_{ut} of 16 – 18 MPa. For bonded magnets with about 60% volume fraction of Nd–Fe–B powder, the PPS bonded magnet shows about twice the tensile strength and half the ultimate strain compared to that of Nylon bonded magnet. At room temperature, the flexural strength is, in general, comparable to the tensile strength. Dynamic elastic modulus measured using the impact resonance method was in good agreement with the elastic modulus obtained from tensile tests. Scanning Electron Microscopy analyses of the fractured surfaces revealed two distinct failure mechanisms. Debonding along the Nd–Fe–B particle and PPS interface is the main cause for failures at 100 and 180 °C. The fracture of Nd–Fe–B particle was observed on the fracture surface of specimens tested at -40 and 23 °C.

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1. Introduction

Since the Nd₂Fe₁₄B-based rare-earth magnetic materials were first reported in the 1980s [1,2], much research has been devoted to the improvement of intrinsic material properties and development of cost-effective fabrication techniques. The Nd–Fe–B permanent magnets have been used to convert mechanical energy to electrical energy and vice versa for various applications [3,4]. In general, Nd–Fe–B magnets can be categorized into two categories: metallic and bonded [5]. The metallic Nd–Fe–B magnets are either produced by the conventional alloy casting and powder metallurgy method [2] or by the hot deformation routes [6]. The bonded Nd–Fe–B magnets can be produced by mixing

the magnetic Nd–Fe–B powder with polymer resins and followed by a molding process. Compression molding, injection molding, extrusion and calendaring are four commonly used molding methods for producing bonded magnets. Among them, injection molding offers the attractive features for mass production and for advanced applications.

One of the advantages of injection molding bonded magnets is the potential to cost-effectively mass-produce the net-shape geometry with accurate dimensional tolerances. Moreover, polymer binders, if properly selected, can also serve as an insulator to isolate the magnetic particle from exposure to the environment and act as a protection against possible corrosion.

Polyphenylene-sulfide (PPS) and polyamide (Nylon) are two commonly used polymer binders for bonded Nd–Fe–B magnets. Nylon has been used in plastic molding for many decades and its basic characteristics

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as well as processing parameters have been established. However, the low melting temperature characteristics limits Nylon-bonded magnets for elevated temperature applications [7]. Unlike Nylon, which melts at about 170 °C (depending on the composition) [8], the PPS exhibits a higher melting temperature of approximately 280 °C [8,9]. The differences in polymer structure, molecular weight, and melting temperature enable PPS-bonded magnets to be used for higher operation temperatures. Furthermore, the low moisture absorption characteristics, good corrosion resistance, high dimensional stability, light weight for metal replacement, and multiple component integration capability all make PPS bonded magnets favorable for many automotive and high temperature applications.

In addition to magnetic properties, mechanical properties of bonded magnets from cryogenic to elevated operating temperatures are also important to ensure the structural integrity and reliability. However, mechanical properties of bonded Nd–Fe–B magnets are not readily available in literature. To broaden the use of bonded magnets, it is essential to establish the basic mechanical characteristics from cryogenic to the desired operating temperatures. Tensile and flexural bending strengths as well as elastic modulus are considered to be the most fundamental characteristics describing mechanical properties of materials, and are widely used in modeling and comparison with other materials.

For sintered Nd–Fe–B magnets, the tensile strength [10], bending strength [11], and fracture toughness [12] have been reported. For bonded magnets, Ikuma et al. [13] have measured the shear strength of extrusion-molded Nd–Fe–B magnets, Garrell et al. [7] have studied the tensile and bending strengths of Nylon bonded magnets, and Xiao and Otaigbe [14] have investigated the effect of liquid crystal polymer and surface modification on mechanical properties of PPS bonded magnets.

The volume fraction of Nd–Fe–B powder in the bonded magnet plays a critical role in determining the magnetic properties. A higher volume fraction of Nd–Fe–B powder usually results in a higher remanence magnetization (B_r) and maximum energy product $(BH)_{\max}$ and are desirable from the magnetic perspectives. However, the higher volume fraction of Nd–Fe–B powder may change the rheology of polymer melt during process and, subsequently, impact the mechanical strengths of bonded magnets. The impact of powder volume fraction to the mechanical strength of finished magnets has seldom been discussed in literature. How to balance the magnetic properties with their corresponding mechanical strengths becomes an important subject for bonded magnet applications.

In this paper, tensile and flexural strengths of PPS bonded Nd–Fe–B magnets with volume fraction of 50–61% are studied from –40 to 180 °C. The dynamic

elastic modulus at room temperature is also discussed. These results are compared with those of selected Nylon bonded Nd–Fe–B magnets.

2. Magnetic aspects of bonded magnets

Prior to entering into the mechanical aspects of bonded magnets, it is essential to discuss a few factors important to material selection, processing, and applications. As mentioned earlier, the volume fraction of magnetic Nd–Fe–B powder plays a critical role in determining the magnetic performance of bonded magnets. Four volume fractions, namely 50, 55, 61 and 69%, were selected using a commercially available MQP-O powder to demonstrate the impact of volume fraction of Nd–Fe–B powder on the magnetic properties. The difference in polymer characteristics and their rheological behavior dictate the maximum volume fraction can be achieved. Volume fractions of 50–55% can readily be obtained for PPS bonded magnets. With some efforts, a volume fraction of 61% can barely be achieved on PPS bonded magnets. This also represents the upper limits of volume fraction for PPS bonded magnets. In comparison, a volume fraction of as high as 71% can be obtained on Nylon bonded magnets.

2.1. Basic magnetic properties at 23 and 180 °C

Shown in Fig. 1(a and b) are the second quadrant demagnetization curves of bonded Nd–Fe–B magnets at 23 and 180 °C. Table 1 summarizes the B_r , H_{ci} , H_c and $(BH)_{\max}$ of PPS and Nylon bonded magnets. As can be seen, all magnets exhibit an intrinsic coercivity (H_{ci}) of 13.3 kOe at 23 °C. As expected, the B_r and $(BH)_{\max}$ values increase with increasing volume fraction. When heated to 180 °C, both B_r and H_{ci} decrease to lower values. Higher B_r values can still be observed on magnets of higher volume fraction. It should be noted that the Nylon bonded magnet at 180 °C has been omitted in Table 1 because the magnet softened and could not maintain its structural integrity to complete the magnetic measurement. The PPS bonded magnet at 69 vol.% is also omitted in Table 1 due to the limitation in molding to achieve such high volume fraction. Although higher volume fraction limits can be achieved on Nylon bonded magnets, the relatively low melting temperature of Nylon hinders the Nylon bonded magnets for high temperature applications. Despite the constraint on lower maximum volume fraction, PPS magnets can sustain the 180 °C applications.

2.2. Flux aging loss

Another important factor influencing the material selection for bonded magnet applications is the flux

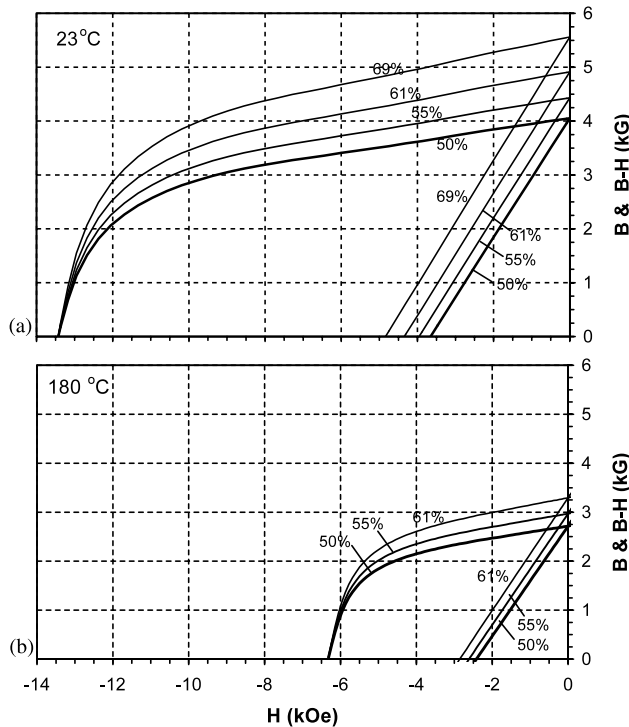


Fig. 1. Magnetic properties of bonded magnets at four different vol.% of Nd–Fe–B particle at 23 and 180 °C (MPQ-O Nd–Fe–B powder produced by Magnequench).

aging loss—the flux loss a magnet encounters when exposed to an elevated temperature for a predetermined duration of time. Fig. 2(a and b) compare the flux aging loss of PPS and Nylon bonded magnets with a length to diameter ratio (permeance coefficient) of 2.0 and volume fraction of 59%. Exponential decrease in flux is observed on all magnets when the magnets are exposed to elevated temperatures. For a fixed aging temperature, the flux aging loss increases, more or less, exponentially with aging duration. For a fixed aging duration, the flux aging loss, in general, increases with aging temperature. Table 2 summarizes the flux aging loss of PPS and Nylon bonded magnets at 100, 125, 150, and 175 °C for 2000 h. Again, the flux aging loss of Nylon bonded magnets at 175 °C was omitted because the magnets could not maintain their structural integrity for a

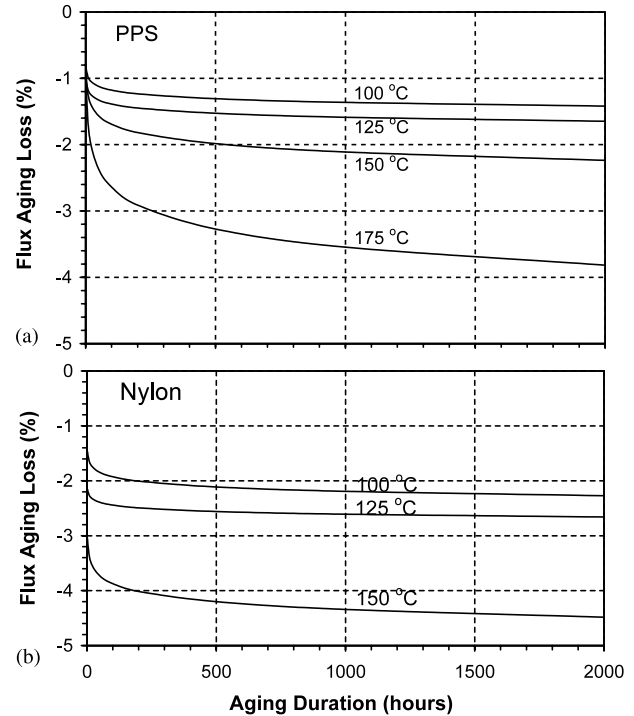


Fig. 2. Flux aging loss of PPS and Nylon bonded Nd–Fe–B magnets at different testing temperatures.

complete aging loss measurement. Table 2 shows that PPS bonded magnets exhibit lower flux aging losses when compared to Nylon bonded magnets with the same dimensions and under the same aging conditions. PPS bonded magnets are preferable if the application requires a lower flux aging loss.

3. Experimental procedure

ASTM (American Society of Test Methods) standards D638-99 [15] and D790-99 [16] were used to determine the tensile and flexural bending strengths, respectively. The test specimens, with dimensions defined by the respective ASTM standards, were prepared by injection molding in two major steps. First, the MQP-O type Nd–Fe–B powder was mixed with PPS,

Table 1

B_r , H_{ci} , H_c , and $(BH)_{max}$ of bonded magnets at volume fraction of 50, 55, 61, and 69 vol.% at 23 and 180 °C

Temperature (°C)	Volume fraction (%)	B_r (kG)	H_{ci} (kOe)	H_c (kOe)	$(BH)_{max}$ (MGoe)	Remarks
23	50	4.03	13.3	3.75	3.72	PPS and nylon
	55	4.41	13.3	4.05	4.37	PPS and nylon
	61	4.89	13.3	4.35	5.36	PPS and nylon
	69	5.53	13.3	5.03	6.71	Nylon only
180	50	2.85	6.47	2.49	1.80	PPS
	55	3.11	6.47	2.69	2.13	PPS
	61	3.45	6.47	2.94	2.58	PPS
	69	NA	NA	NA	NA	PPS

lubricant, and other additives according to a predetermined mixing ratio to achieve the targeted volume fractions. The mixture was then compounded and pelletized into small pieces as feedstock for injection molding. The injection molding was performed under an argon atmosphere.

The tensile tests were performed on a MTS 808 universal testing machine. The standard Type-IV specimen specified in ASTM D638-99 [15] with 3.2 mm by 6 mm rectangular test cross-section was used. The crosshead was controlled at a speed of 0.02 mm s^{-1} and two hydraulic grips were used to apply a constant 1.5 MPa pressure on the specimen. Tests were carried out at four temperatures, namely -40 , 23 , 100 , and 180°C .

The room temperature tests were carried out at 23°C with a controlled room humidity of 50% RH. A load-cell with a capacity of 1.4 kN was used to monitor the applied load. As shown in Fig. 3, an Applied Test System (ATS) furnace was used in combination with an ATS PID temperature controller for cryogenic and elevated temperature tests. For cryogenic tests, the ATS furnace was connected to a liquid nitrogen tank and set to -42°C . A type K thermocouple was placed next to the specimen surface to monitor the actual sample temperature. Samples were soaked for 10 min before the initiation of the mechanical test. The crosshead speed was set to 0.01 mm s^{-1} .

For tests at 100 and 180°C , the ATS controller was set at slightly higher furnace temperatures of 113 and 195°C , respectively, which were determined by comparing the temperature recorded by a thermocouple embedded in a dummy test specimen. Samples were soaked for at least 20 min before each test to assure uniform temperature distributions of 100 and 180°C throughout the specimen. The crosshead was moved at a speed of 0.02 mm s^{-1} .

Table 2

Comparison of the flux aging loss of PPS and Nylon bonded magnets with MQP-O powder and with volume fraction of 61% after being aged at 100 , 125 , 150 , and 175°C for 2000 h

Aging temperature ($^\circ\text{C}$)	Flux aging loss (%)	
	PPS-bonded	Nylon-bonded
100	1.41	2.38
125	1.63	2.71
150	2.21	4.55
175	3.80	NA

The four-point flexural bending tests following ASTM standard D790-99 [16] were performed on a MTS 120 testing machine at room temperature. The crosshead speed was 0.01 mm s^{-1} . The specimen dimensions were $125.7 \text{ mm} \times 6.4 \text{ mm} \times 12.7 \text{ mm}$.

The elastic modulus was tested according to ASTM Standard C1259-98 [17]. The resonant frequency in the flexural mode of vibration was measured and used to calculate the dynamic elastic modulus. The test was conducted at room temperature and the specimen dimensions were the same as the flexural bending test.

Table 3 lists the sample codes, corresponding volume fraction and type of polymer binder for bonded Nd–Fe–B specimens. Complete tensile tests across four different temperatures were conducted on PPS503 and PPS517. As discussed in Section 2, due to the limitation of about 61 vol.% of Nd–Fe–B powder for molding PPS bonded magnets, only a limited number of PPS614 tensile specimens, enough for the room temperature tensile test, were produced. The PPS614 specimens with larger cross-section for bending and dynamic elastic modulus tests were successfully produced and tested. Test results of two Nylon samples, Ny597 and Ny710, were also presented for comparison.

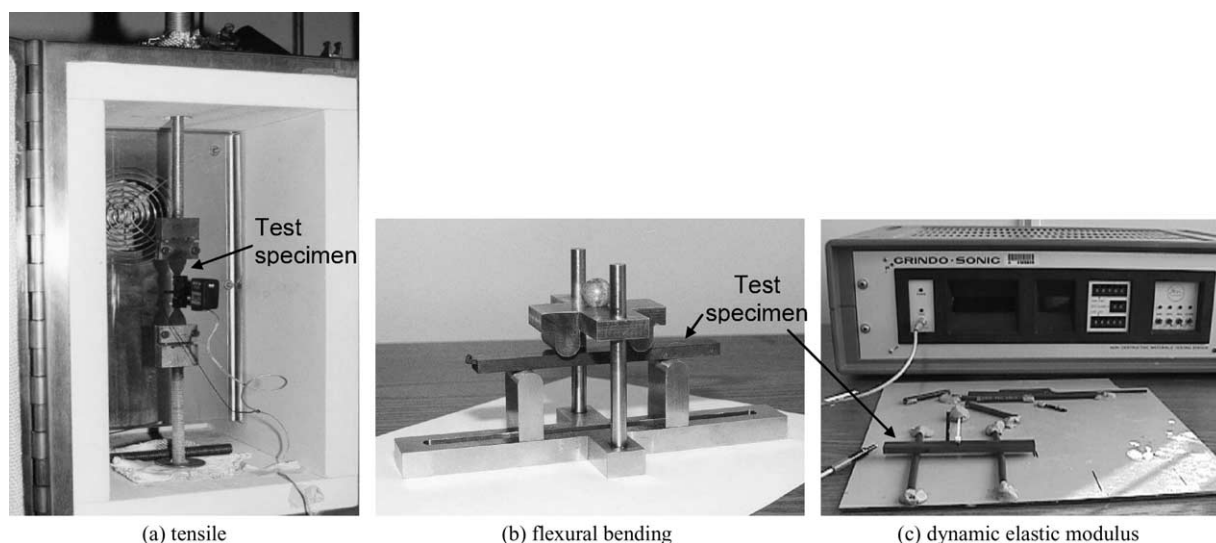


Fig. 3. Schematics of the test setup.

Table 3
Sample designation and corresponding volume fraction of PPS and Nylon bonded Nd–Fe–B magnets

Numbering scheme	Polymer type	Estimated volume fraction (%)
PPS503	PPS	50.3
PPS517	PPS	51.7
PPS614	PPS	61.4
Ny597	Nylon	59.7
Ny710	Nylon	71.0

4. Results and discussion

4.1. Tensile

Fig. 4 shows the tensile stress–strain curves for three PPS (PPS503, PPS517, and PPS614) and two Nylon (Ny597 and Ny710) bonded magnets at room temperature. The strain was measured using an extensometer. 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. For both PPS and Nylon, the increase in volume fraction of Nd–Fe–B reduced the ultimate strain in tensile test. With an ultimate strain of less than 0.6%, all five types of bonded magnets exhibited very limited ductility. Moreover, the ultimate tensile strength (S_{ut}) of PPS bonded magnets decreases with the increasing volume fraction of Nd–Fe–B powder as evidenced by PPS503, PPS517, and PPS614. The decrease in S_{ut} may be explained by the fact that the ultimate tensile strain decreases with the increasing volume fraction of Nd–Fe–B powder. Despite the decrease in ultimate tensile strain, the S_{ut} of Ny710 is higher than that of Ny597 suggesting the S_{ut} of Nylon bonded magnets may increase with increasing volume fraction of Nd–Fe–B powder. This opposite trend in ultimate tensile strain

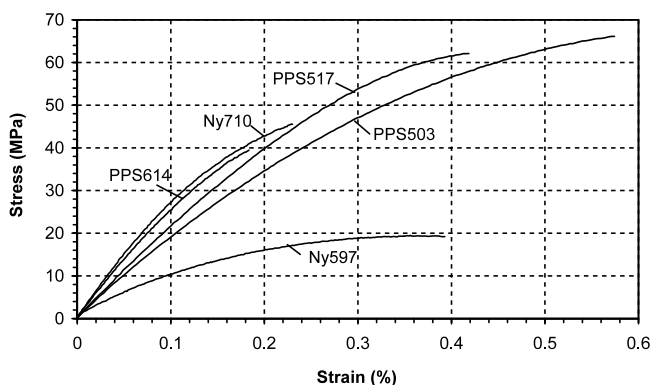


Fig. 4. Tensile stress–strain curves of four injection molded Nd–Fe–B magnets at 23 °C.

and S_{ut} may be caused by the different polymeric nature between PPS and Nylon: PPS is more brittle and Nylon is more ductile.

Comparing PPS614 and Ny597, the difference in powder volume fraction is very limited. However, the S_{ut} of PPS614 is about twice higher than that of Ny597, even though the ultimate tensile strain of PPS614 (1.8%) is only half the value of that of Ny597 (3.9%). This suggests that the tensile strength of PPS bonded magnets is higher than that of Nylon magnets at a given tensile strain for a fixed Nd–Fe–B powder volume fraction. Again, this difference can be explained by the difference in polymeric structure and their response under tensile conditions. PPS bonded magnets also have a higher elastic modulus, which will be further verified (see Section 4.3) using the dynamic impulse elastic modulus measurements.

Fig. 5 summarizes the variation of tensile strength, S_{ut} , of two PPS bonded magnets at –40, 23, 100, and 180 °C and the two Nylon bonded magnets at –40, 23, 100 °C. Cooling to –40 °C enhances the S_{ut} for both Nylon and PPS bonded magnets. The tensile strength of two PPS bonded magnets at –40 °C increases by about 20% relative to that at room temperature. This can be explained by the transition of the PPS and Nylon bond to a more brittle, glassy phase at lower temperatures. At cryogenic temperature, the difference in thermal expansion coefficient between PPS ($5.3 \times 10^{-5}/^{\circ}\text{C}$) and Nd–Fe–B ($4\text{--}8 \times 10^{-6}/^{\circ}\text{C}$) [18,19] generates a compressive residual stress on the Nd–Fe–B powder by the surrounding PPS bond. This compressive residual stress on the interface between the PPS bond and Nd–Fe–B particles may help improve the tensile strength of PPS bonded magnets at cryogenic temperature. The same phenomenon of increasing tensile strength at cryogenic temperature due to the transition of bond material characteristics and the difference in thermal expansion coefficient has also been observed in Nylon bonded magnets [7].

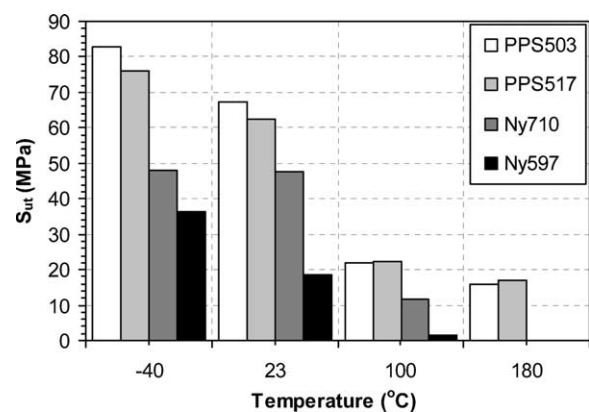


Fig. 5. Tensile strength of the two PPS bonded injection molded samples at –40, 23, 100, and 180 °C in comparison with Nylon bonded magnets at –40, 23, and 100 °C.

Although the PPS bonded magnets soften considerably at 100 and 180 °C, as shown in Fig. 5, the S_{ut} remains in the 16–18 MPa range. This suggests that these materials can maintain the structural integrity for high temperature applications. In comparison, Nylon melted and was not able to withstand the 180 °C temperature required for some automotive applications [7].

Fig. 6 shows the tensile stress–strain curves of PPS503 at three testing temperatures. The typical structure for PPS is a mixture of amorphous and crystalline phases (semicrystalline). Two distinct trends of stress–displacement curves are obtained for data taken at 23 °C and at 100 and 180 °C. The glass transition temperature, T_g , for PPS is about 85–90 °C [9,19]. For the test temperature above T_g , i.e. 100 and 180 °C, the amorphous phase starts to soften and causes the reduction in strength. The crystalline phase softens at a higher temperature. The strain hardening effect due to chain alignment at larger strain (14%) observed in Nylon bonded magnets with spherical shape magnetic particles [7] is not seen in Fig. 6 because of the low ductility. The irregular plate shape of the magnetic particles in the bonded magnets used in this study creates localized stress concentration and limits the magnitude of strain to fracture. On samples tested at 100 and 180 °C, strain in the level of 1.4–1.6% was obtained. It is noted that strain reported in Fig. 6 was obtained from the cross-head displacement without a machine compliance correction. This effect is not significant under the small loads for testing of bonded magnets.

4.2. Flexural bending

Fig. 7 shows the stress–displacement curves for the bending test of PPS517, PPS614, and Ny597 at 23 °C. The displacement is measured at the set of two inner loading pins, as shown in Fig. 3(b), in the four-point

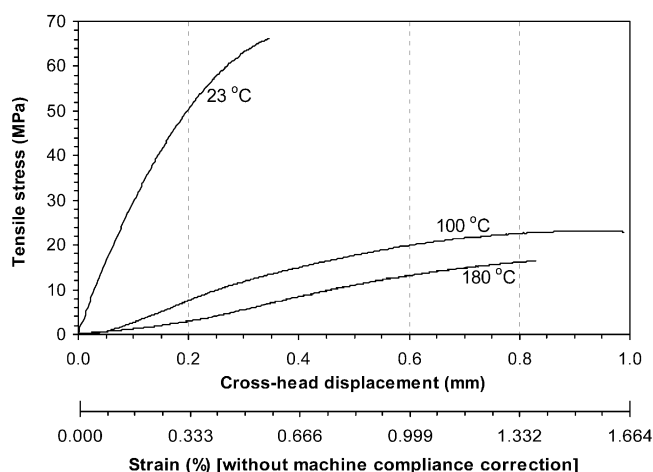


Fig. 6. Tensile stress–strain curves of PPS503 at 23, 100, and 180 °C.

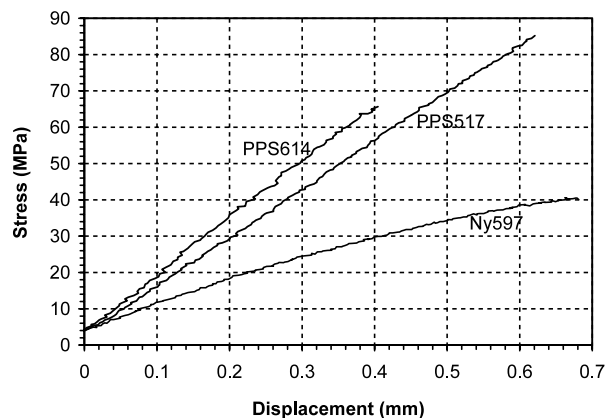


Fig. 7. Flexural bending strength of the two PPS and one Nylon bonded Nd–Fe–B magnets.

bending test configuration. The bending stress is calculated from the measured load.

$$\sigma_{\max} = \frac{3PL}{2bd^2}, \quad (1)$$

where P is the load, L ($= 38.1$ mm) is the difference in span of the supporting and loading roller sets, b ($= 12.7$ mm) is the specimen width, and d ($= 6.4$ mm) is the specimen thickness. Seven bending tests were performed for each material and the averaged results are presented in Fig. 7. The sample standard deviation for the strength at any strain across the test range did not exceed 4.97 MPa. This indicates that the samples were of uniform quality and the tests were reproducible.

The bending strengths of PPS517 and PPS614 are 85 and 65 MPa, respectively. These values are higher than their corresponding tensile strengths, 63 and 40 MPa for PPS517 and PPS614, respectively. Due to the non-uniform loading and smaller volume of material subjected to the peak stress in bending, such trend is commonly observed in comparing the strength results in bending and tensile tests. The displacement to fracture for PPS614 (0.41 mm) is shorter than that of PPS517 (0.63 mm). A similar trend of limited strain to fracture has been observed in the tensile test results in Fig. 4. The higher volume fraction of Nd–Fe–B powder makes the PPS bonded magnet more brittle.

4.3. Dynamic elastic modulus

Ten frequency readings of PPS517 and PPS614 specimens were recorded to measure the dynamic elastic modulus following the ASTM procedure [17]. These readings fell within a standard deviation of 1.5 MPa, which showed the consistency of the experimental measurements. The dynamic elastic modulus was then calculated using the following equation [17]:

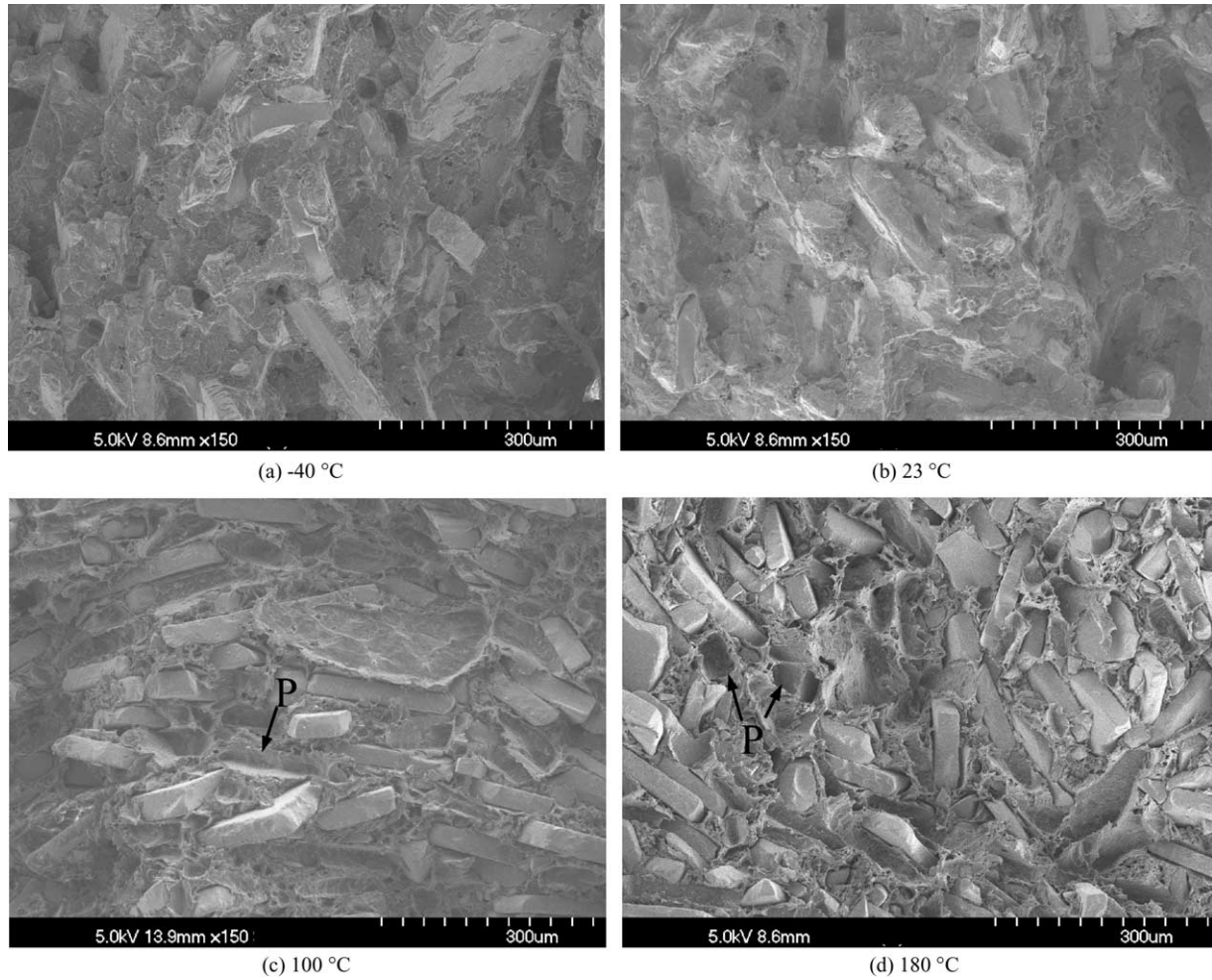


Fig. 8. SEM micrographs of fractured surface of PPS517 at -40 , 23 , 100 , and 180 °C.

$$E = 0.9465 \left(\frac{mf_f^2}{b} \right) \left(\frac{L^3}{t^3} \right) T_1, \quad (2)$$

where E is the elastic modulus, m is the mass of bar in g, b is the width of bar in mm, L is the length of bar in mm, t is the thickness of bar in mm, f_f ($= 2,000,000/R$) is the fundamental frequency of the bar in flexure in Hz, R is the reading from Grindo-Sonic testing machine, and T_1 is the correction factor as to be determined according to the ASTM Standard [17]. Different values for Poisson's ratio, ranging from 0.2 to 0.4, were assumed to calculate the elastic modulus. It was found that the Poisson's ratio contributes minimally to the results of elastic modulus.

The dynamic elastic modulus of PPS517 and PPS614 were determined to be 24.1 and 31.7 MPa, respectively. These values are very close to the elastic modulus of 25.5 and 31.8 MPa determined by the tensile tests. The difference in elastic modulus measured by these two methods is 5.5 and 0.5% for PPS517 and PPS614, respectively. These results indicate that the dynamic

impact resonant test is a good non-destructive testing method for measuring the elastic modulus and can possibly be used to detect the flaws and defects in bonded magnets. The results also show the effect of higher vol.% of Nd–Fe–B powder. The elastic modulus of PPS614 is about 24% higher than that of PPS517.

Compared to the 12–16 GPa elastic modulus for Nylon bonded magnets [7], the PPS bonded magnets have a higher elastic modulus and will exhibit smaller deformation under the same level of load.

5. SEM micrographs of fractured surfaces

Scanning Electron Microscopy (SEM) was used to examine the fracture surfaces and gain insight into the failure mechanism of PPS bonded magnets. Fig. 8 shows the fracture surfaces of PPS517 at four testing temperatures. SEM micrographs of fracture surface of Ny597 at 23 and 100 °C are shown in Fig. 9 for cross-references.

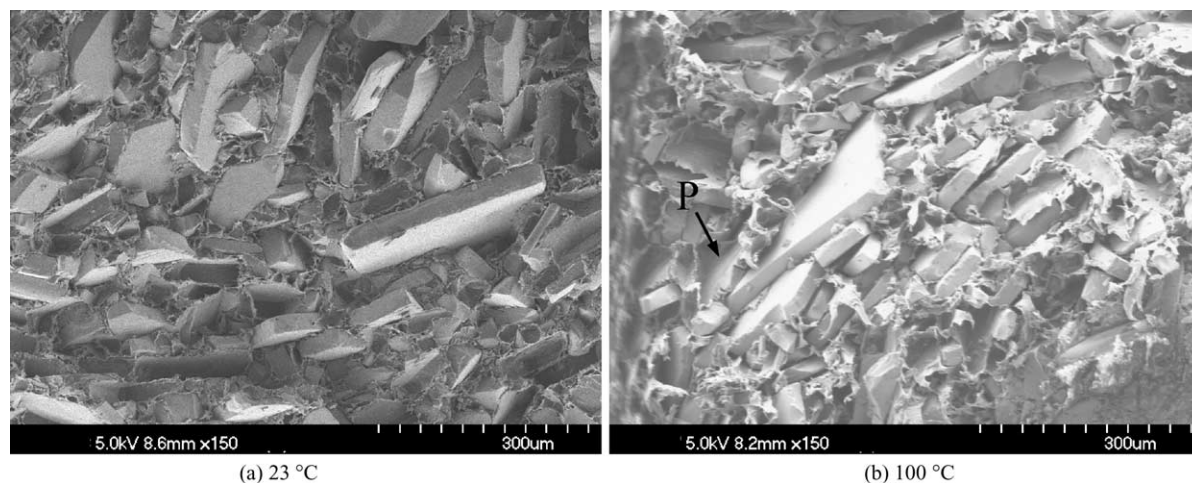


Fig. 9. SEM micrographs of fractured surface of Nylon bonded Ny597 at 23 and 100 °C.

Below the glass transition temperature, as shown in Fig. 8(a) for specimens tested at -40 °C and Fig. 8(b) for specimens tested at 23 °C, the PPS bonded magnet exhibits brittle behavior, the fracture surface is glassy, and the features of the plate-shaped Nd–Fe–B powder are not easily recognizable. In comparison, for the PPS517 specimen evaluated at 100 and 180 °C as well as the Ny597 specimen evaluated at 23 and 100 °C, the irregular plate shape of the Nd–Fe–B particulates is apparent on the fracture surface. The pocket left by the Nd–Fe–B particle that pulled-out, marked as P, can also be observed. At the highest testing temperature of 180 °C for PPS517 and 100 °C for Ny597, the matrix is softened and residual burr or flash can be identified on the fracture surface between Nd–Fe–B particles.

The difference in surface morphology indicates that Nd–Fe–B particles ruptured or fractured at -40 and 23 °C for PPS517. This leaves the glassy surface without apparent pull-out pockets and plate-shape Nd–Fe–B particle. Because the elastic modulus of the magnetic particulates is higher than that of the surrounding matrix, tensile loads in the matrix are transferred by shear of magnetic particles. The rate of load transfer is proportional to the interfacial shear stress and when the magnitude of this stress is high, and the applied external load to the composite is also high, the reinforcing particles will fracture before they can redistribute their load back to the matrix. The strength of the Nd–Fe–B particle also helps to increase the tensile and bending strength of material at -40 and 23 °C, as indicated in Figs. 5 and 6. On the contrary, the debonding on the surface between Nd–Fe–B particle and PPS matrix is the major source for failure for PPS517 at 100 and 180 °C. At high temperature, the Nd–Fe–B particle did not fracture due to poor bonding to the matrix and the overall strength of bonded magnets was reduced.

6. Concluding remarks

The data presented in this paper pertaining to the mechanical properties of injection molded Nd–Fe–B/PPS type magnets offers information which will be useful in the selection and design of bonded magnets depending on their determining property and operating conditions. The PPS614 exhibits about twice the tensile strength and half the ultimate strain compared to that of the Ny597, which has about the same level of volume fraction of the Nd–Fe–B powder as the PPS614. The PPS bonded magnets showed an overall higher strength than Nylon bonded magnets, particularly at elevated temperatures. This research quantified the mechanical strength of PPS bonded Nd–Fe–B magnets at 180 °C and demonstrated its capability to maintain structural integrity. The dynamic elastic modulus of PPS bonded Nd–Fe–B was measured and found to be very close to the value obtained from conventional tensile tests.

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