THERMAL EXPANSION AND VISCOELASTIC PROPERTIES OF SINTERED POROUS FERROUS COMPONENTS

A.Antonyraj, Seong Jin Park and Randall M.German

Center for Advanced Vehicular Systems, Mississippi State University,
200 Research Blvd., Starkville, MS 39759, USA

ABSTRACT

A method of studying thermal expansion and viscoelastic properties of sintered porous ferrous components is presented. In this presentation, iron compacts in the range of 68 to 87% dense were prepared and the change in thermal expansion and its coefficient with temperature is studied using dilatometer. The porous samples were also subjected to cyclic load by varying the frequency, ranging from 50 Hz to 1 Hz as a function of temperature with range of 25 °C to 280 °C using a dynamic mechanical analyzer (DMA). Based on the experimental results we developed a model for thermal expansion and viscoelastic properties, which can be used in finite element simulations. Such formal developments are critical to future computer simulations underpinning dynamic applications for powder metallurgy (PM).

INTRODUCTION

Materials possessing quality associated with long durability and stability under abnormal environments such as acoustic absorption, mechanical damping and energy absorption are used in both structural and nonstructural usage. In these prospects, porous metals and ceramics are being considered. Some of the applications include are aircraft passenger seats, nuclear fuel transportation, and hazardous material containment [1-6]. It has been considered in many years, the use of heavy porous materials as structural materials offer great strength compare to light weight porous materials due to its great ability to absorb high energy when impact load fall on this material. Based on the literature survey, research on thermal expansion and viscoelastic behavior of bulk polymeric materials and their composites are well advanced, compared to PM components. Prediction of densification behavior from finite element sintering simulation using thermal expansion coefficient value and evaluation of performance of sintered parts from structural simulation using viscoelastic data can easily be made. Hence it is of great importance to investigate the thermal expansion and the dynamic mechanical behavior of PM components made from powder metal.

In the present study, the thermal expansion and the dynamic mechanical behavior of sintered porous iron premix compacts, possessing densification in the range of 68 to 87 %, as a function of temperature and frequency by using dilatometer and dynamic mechanical analyzer (DMA) are studied. In addition, Rockwell hardness, to establish a relationship between the effect of hardness and porosity, is also measured. For this, the powder was compacted under various pressures, debound and then presintered.

EXPERIMENTAL CONDITIONS

POWDER CHARACTERISTICS

Material chosen for this investigation is iron premix powder which was supplied by North American Hoganas Inc. with its composition and physical properties, taken from the company material data sheet are furnished in Table I & II. The free flow capability of powder was also determined by using Hall flow apparatus which took 25 seconds for 50 g flow of iron premix powder.

Table I. Chemical Compositions and Theoretical Density of Powder Metal

Material	Ni (wt.%)	Mo (wt.%)	C-Graphite (wt.%)	Lubricant (wt.%)	Fe (wt.%)
Iron premix	2.06	0.83	0.26	0.73	Balance

Table II. Physical Properties

Flow Hall (+24 h)	25 s / 50 g
Apparent density	3.16 g/cm^3
Theoratical density	7.64 g/cm^3

PREPARATION OF COMPACTS

Specimens were made compaction in a cylindrical die at various pressures (45 MPa to 195 MPa). Depending upon the measurements and easy handling, the dimension of the specimen has been changed according the following. For DMA test we used iron bar of dimension 6 mm wide, 4 mm thick and 37 mm long and for dilatometer test the area of the specimen maintained was 13 mm wide, 4 cm thick and 3.2 cm long. For hardness testing, specimens with well smooth surface were selectively selected.

The relative density of the sample was determined from the change in mass and its dimension before and after compaction by Carver hydraulic press (Figure 1). Then all the specimens were kept in a furnace and heated to 400 °C at the heating rate of 5 °C/min and allowed to dwell for 180 minutes at this peak temperature to burn out all the polymers. The selection of this peak temperature was made from thermogravimetric analysis (TGA) curve (Figure 2) which explored the binder removal region of 190 °C to 360 °C for the experiment carried out at the ramp rate of 5 °C/min using Setsys 2000 system (Setaram Instrumentation).

Sintering was carried out in nitrogen-20% hydrogen atmosphere at a heating rate of 3 °C/min from room temperature (25 °C) to the peak temperature of 800 °C and allowed to stay at this temperature for 60 minutes. Unconditional cooling was preferred to bring down the system temperature from the peak temperature, 800 °C to the room temperature (25 °C). After sintering process, the density was calculated from its mass and dimensions.

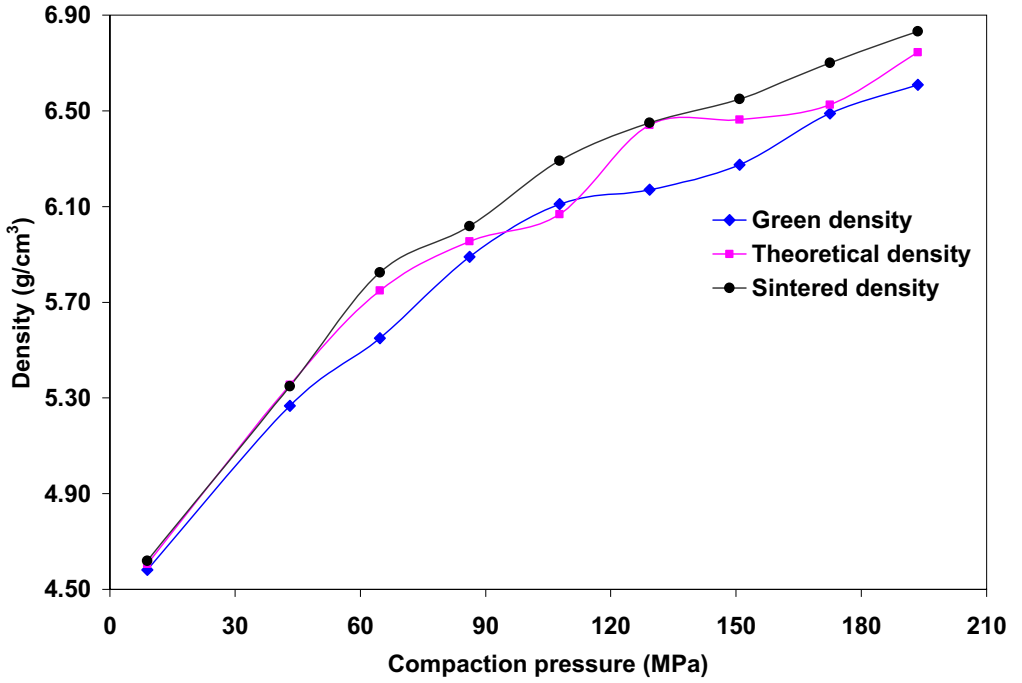

Figure 1. Variation of green, theoretical, and sintered density with compaction pressure.

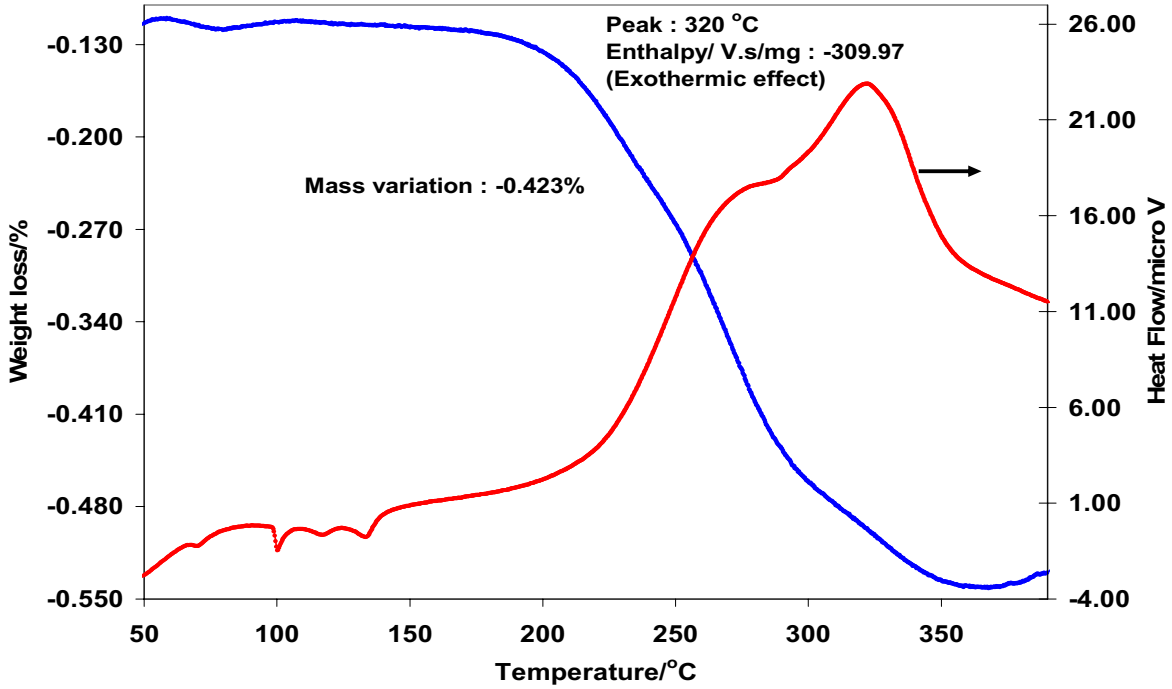

Figure 2. TGA-DSC curve

DILATOMETER

The change in dimension of the specimens was also studied to calculate thermal expansion with function of porosity using vertical push rod dilatometer (Unitherm Model 1161, Anter Corporation) in pure

nitrogen atmosphere. For this test, the constant heating rate of 5 °C/min toward the peak temperature of 800 °C was employed. The dwell time set at the peak was 60 minutes.

ROCKWELL HARDNESS:

To bring the relationship between hardness and density, hardness measurement in Rockwell mode was performed by using Leco Rockwell type hardness tester LR.

DYNAMIC MECHANICAL ANALYZER

To study the mechanical properties under dynamic conditions, we used a dynamic mechanical analyzer, DMA Q800 model (TA Instruments Corp. Pittsburg, USA). Initially the samples were equilibrated at 25 ± 0.1 °C and purged with oxygen (20 ml/min) through air-cooling device. The static and dynamic force set was 700 mN. Tests were conducted to evaluate elastic modulus and damping, as functions of frequency, temperature, and combinations of these two under bending mode. For the temperature scan mode, we kept the frequency of the cyclic load constant at 50 Hz and the temperature was programmed in the range 80 °C to 250 °C. For the frequency scan mode, we kept the temperature constant as 80 °C and the frequency was programmed in the range 50 Hz to 1 Hz. The temperature and frequency dependent behavior was studied by recording the changes in storage modulus (strain) and the phase angle (tangent delta).

RESULTS AND DISCUSSION

DENSITY MEASUREMENT

Figure 1 shows the variation of green and sintered densities. The curve of die compaction behavior is typical as density increases with compaction pressure. Specimen prepared for the application of low compaction pressure of 45 MPa showed the green density value 4.58 g/cm^3 (58 %) and for the specimen prepared for the application of high compaction pressure of 195 MPa showed the green density value 6.61 g/cm^3 (84 %). After presintering, the densities of all specimens increased about 1-3 %. The lowest and highest value of sintered density obtained was 4.62 g/cm^3 (68 %) and 6.83 g/cm^3 (87 %) respectively. For the thermal expansion and viscoelasticity measurement, the range of density is 68 to 87 % and the range of porosity is 11-29 %.

THERMAL EXPANSION

Figure 3 shows the change in thermal expansion of sintered porous iron pellets possessing the densification 68, 82 and 87%. In all the cases the thermal expansion was linearly increased with raise in temperature. Higher thermal expansion value was obtained for the compacts prepared at high densification values which gradually decrease with decrease in densification of compacts. The linear thermal expansion coefficient (CTE) at 300 ^{o}C was calculated to be $6.7 \cdot 10^{-6}$ K^{-1}, $7.2 \cdot 10^{-6}$ K^{-1}, and $10.6.10^{-6}$ K^{-1}. This indicates the thermal expansion coefficient depends on the porosity of the material. This result is far from the following classical model for predicting CTE of porous material

$$\alpha_\rho = \alpha_0 \rho^{1/3} \qquad (1)$$

where α_ρ is CTE of porous material with density of ρ and α_0 is CTE of full dense material. The error range is 18-70 %.

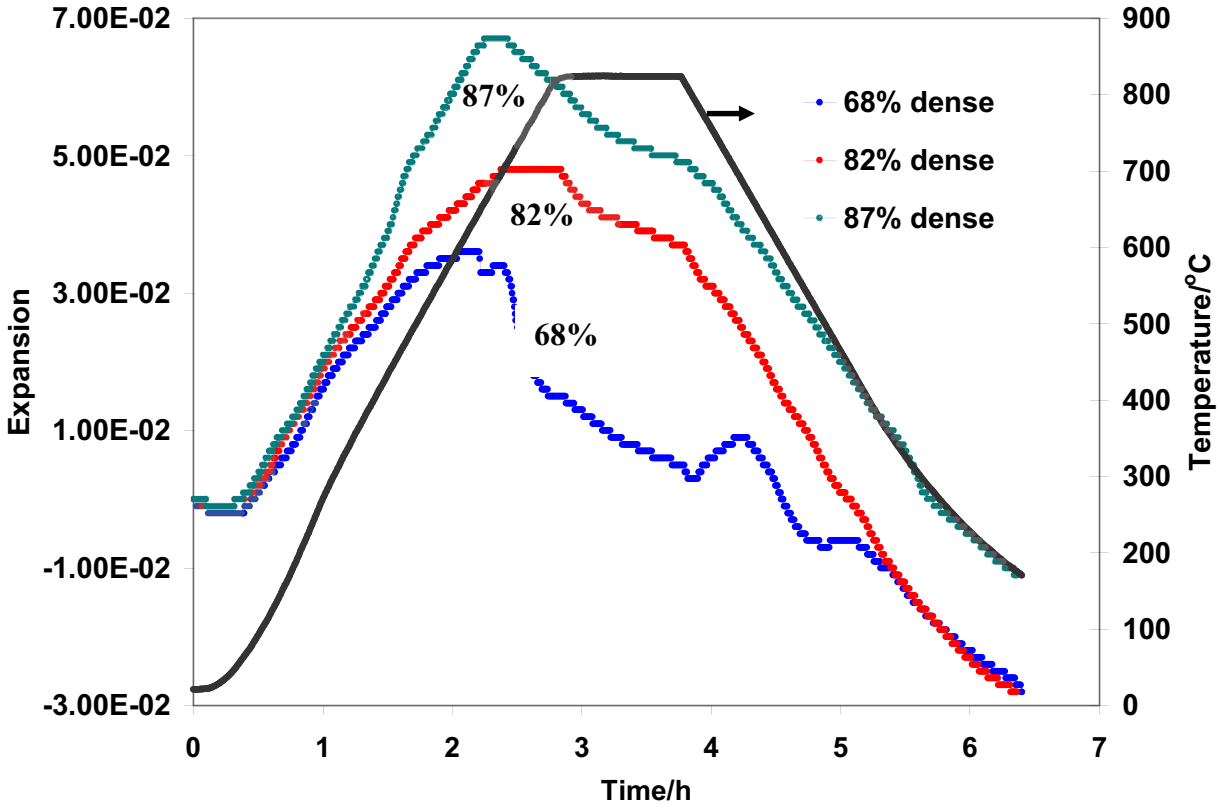

Figure 3. Thermal expansion of sintered porous iron pellets

STORAGE MODULUS AND LOSS MODULUS

Figure 4 illustrates the definition of an elliptical hysteresis loop. An expression given by Dowling [7] in Equation (2) describes the relationship between the storage modulus, E' and loss modulus, E''

$$E^* = E' + iE'' \tag{2}$$

Here i is the square root of -1, the absolute value E^* and Young's modulus E. The second parameter calculated in this study tangent delta ($\tan\delta$) which is the ratio of E'' to E'. δ is the phase angle between the dynamic stress and the dynamic strain. An elliptical hysteresis loop shown in Figure 4 and a sinusoidal stress σ expressed in time domain t with frequency ω and amplitude σ_a given in equation (3) is appropriate for energy dissipation (Δu) calculation.

$$\sigma = \sigma_a \sin \omega t \tag{3}$$

which is assumed to be applied and the strain response ε with amplitude ε_a has a phase shift.

$$\varepsilon = \varepsilon_a \sin \omega t \tag{4}$$

Clearly from Figure.4, we have

$$E^* = \frac{\sigma_a}{\varepsilon_a} \text{ and } E' = E^* \cos\delta \tag{5}$$

Note that

$$\Delta u = \sigma_a \varepsilon_a \sin\delta \tag{6}$$

is the total energy dissipated in each cycle per unit volume of material while the shaded triangular area u_e in Figure 4 is the elastic energy.

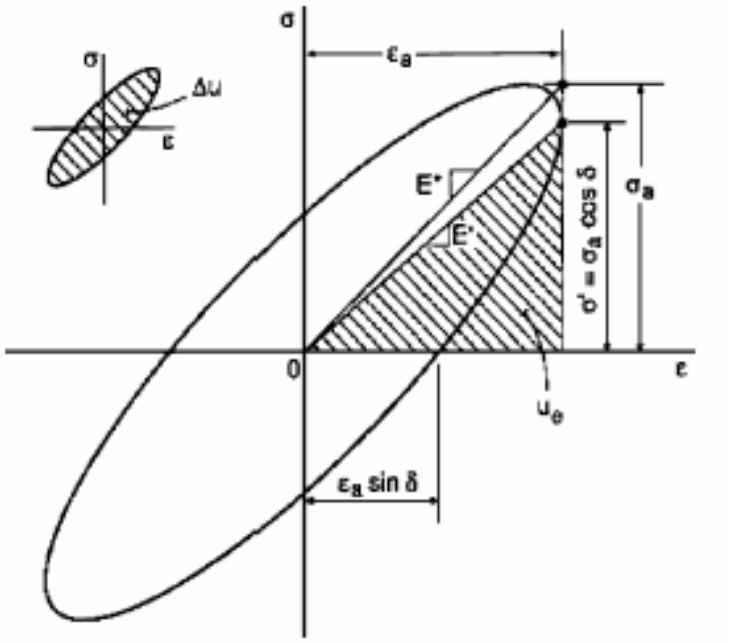

Figure 4. Typical hysteresis loop for viscoelastic materials [7].

The variation of storage modulus, tan delta, and loss modulus with temperature are shown in Figures 5-8. The influence of densification and oscillating frequency is shown in Figure 9. As shown in Figures 5 and 6, the storage modulus exhibited by the test samples was in the range of 7.5 to 34.5 GPa, which greatly depends on density of sample. But the values decreases with increase in temperature during temperature scan mode particularly in the temperature range 80 °C to 250 °C. Further raise in temperature the decrease in storage modulus value was low. At 250 °C, the storage modulus loses about 10 % of original value. There was a point of deflection at about the temperature of 80 to 100 °C. Figure 6 shows the effect of storage modulus with densification value at different temperatures. The storage modulus value increases with increase in densification value. Generally high temperature yields higher storage modulus values and low temperature yield lower values.

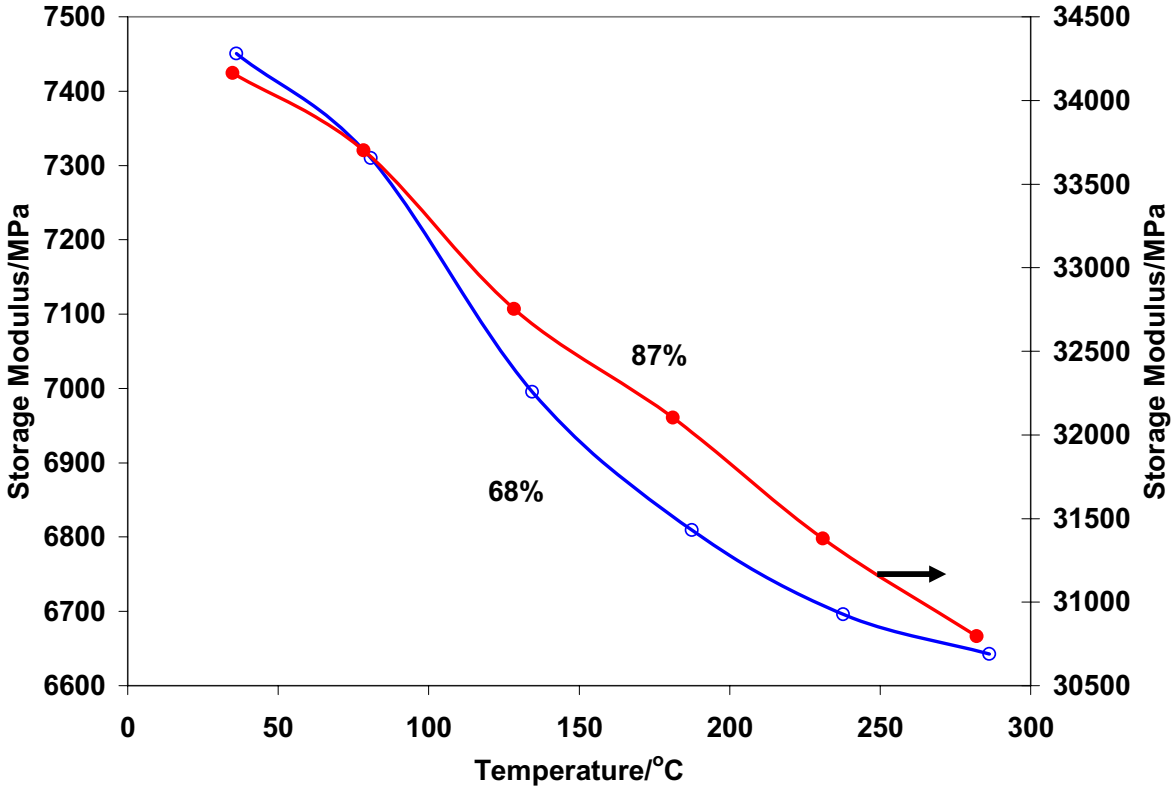

Figure 5. Storage modulus of sintered porous iron pellets versus temperature

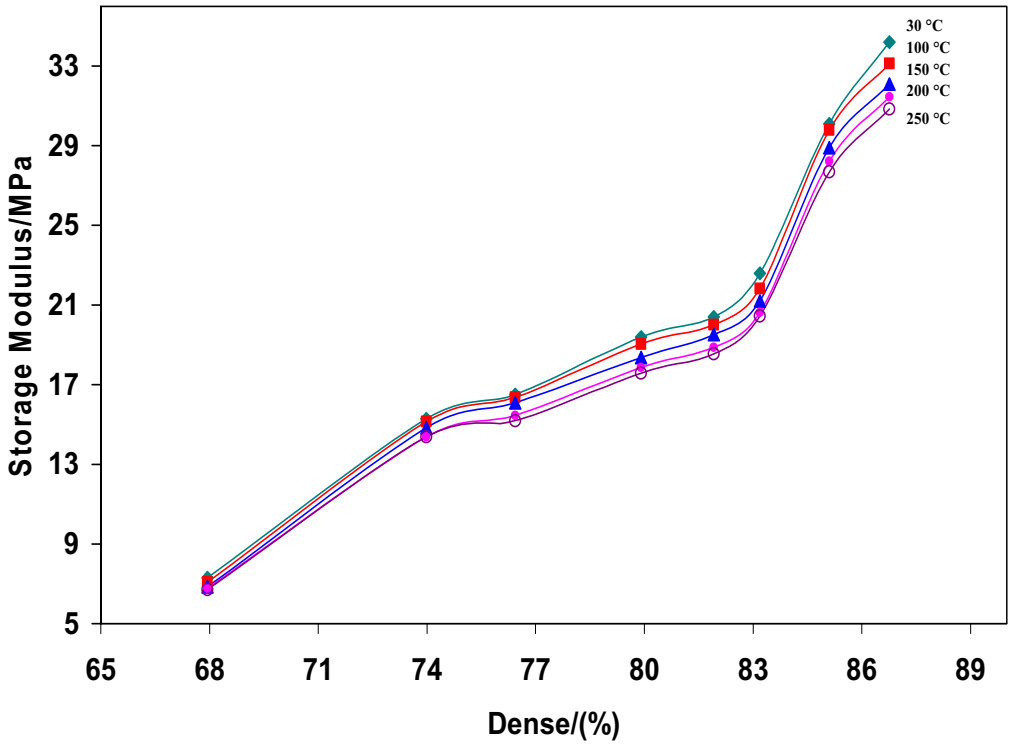

Figure 6. Storage modulus of sintered porous iron pellets versus densification

The tangent delta shown in Figure 7 shows the tangent delta increases rapidly and reaches a maximum and then reduces its value. This peak value is normally referred as a loss peak. The loss modulus (energy dissipation) measured during viscoelastic measurement are shown in Figure 8. Usually the dependence of viscous flow and elastic flow behavior is represented by loss modulus master curve and storage modulus master curve. Figures 5 and 8 establish a relationship between the loss modulus and storage modulus master curves (at a certain frequency) which is a measure of viscoelastic behavior of the system.

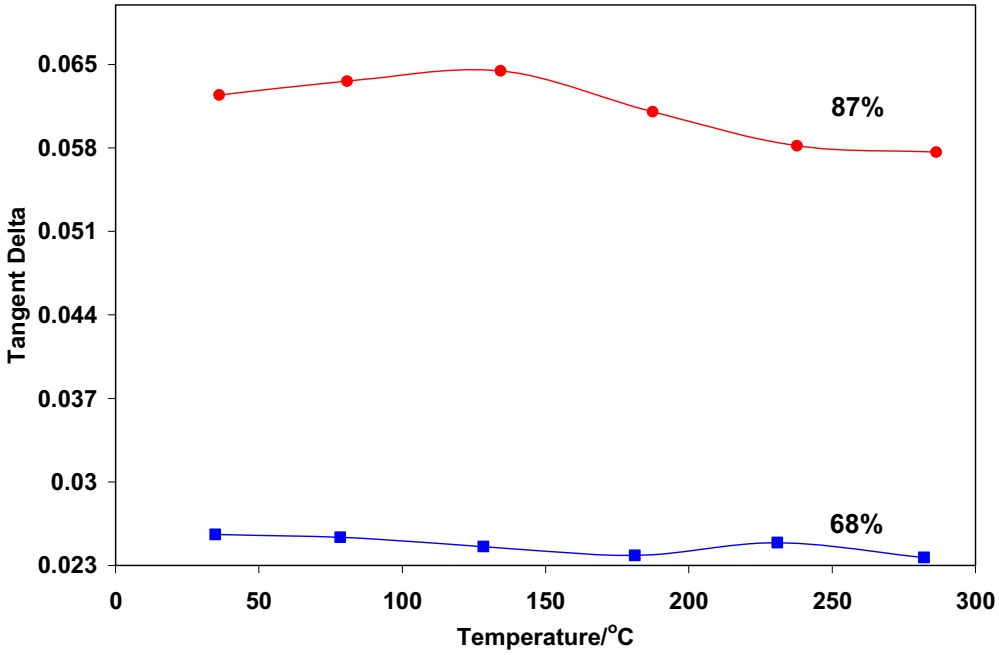

Figure 7. Tangent delta of sintered porous iron pellets versus temperature

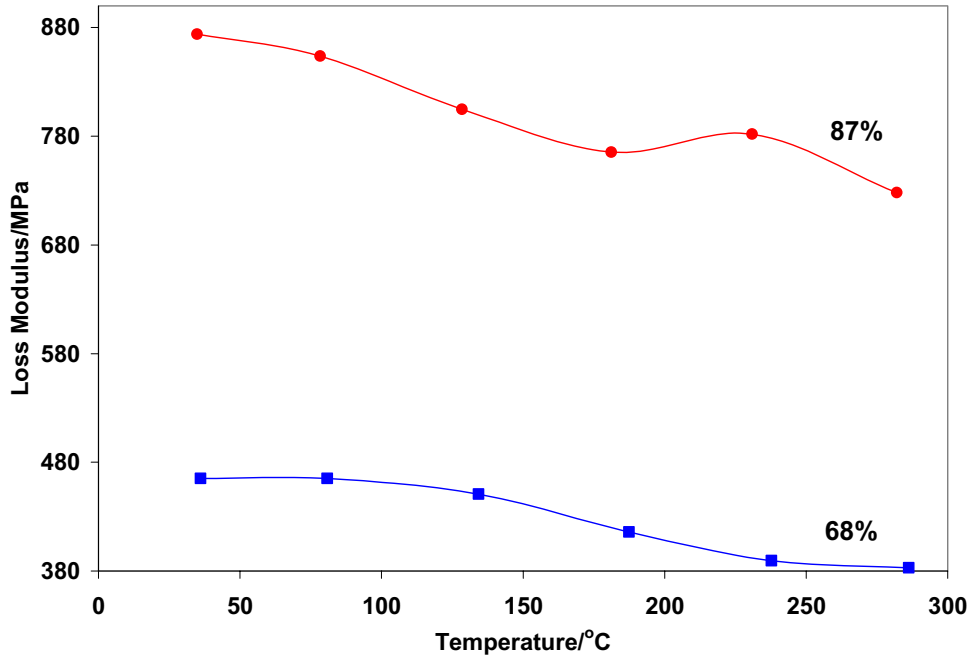

Figure 8. Loss modulus of sintered porous iron pellets versus temperature

The frequency dependence on storage modulus is shown in Figure 9. Typically this behavior is similar to a temperature scan mode in which the storage modulus decreases gradually.

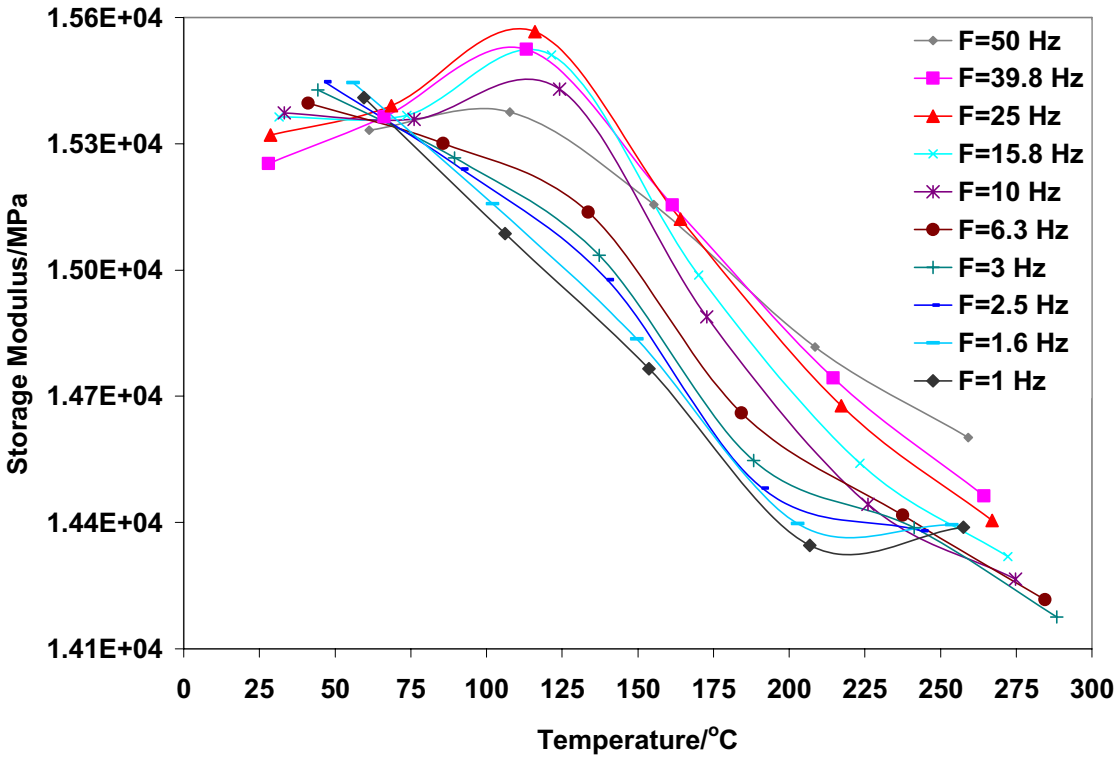

Figure 9. Variation of storage modulus with various frequency of oscillation for the compact prepared at the densification value of 74%

HARDNESS

Rockwell hardness measurements revealed that the hardness values gradually decreases with decrease in sintered density value. Compacts prepared at high densification values of 87% showed 22-23 whereas compact prepared at the densification value 83% showed 15-16 for the measurements made in HRG scale.

CONCLUSIONS

Test conducted on 3-point bending mode revealed that there is a great influence on storage modulus, loss modulus and tangent delta with temperature and frequency. From these parameters one could understand the energy dissipation and damping capacity of material under dynamic loading within the elastic limit. Expansion of iron pellets possessing the densification 87% is higher than that of 68%.

REFERENCES

1. M. Kellomaki, J. Astrom and J. Tomonen, *Phys. Rev. Lett.* 77 (1996) 2730
2. V. Shaplvalov, MRS Bull., (1994) 24.
3. H. Fusheng, Z. Zhengang and L. Changsong, *Scr. Metall. Mater.* 37 (1997) 1441.

4. H. Fusheng, Z. Zhengang and L. Changsong, *Acta Phys. Sin (in Chineese)* 47 (1988) 372.
5. B. Wang, J. R. Klepaczko, G. Lu and L. X. Kong, "Viscoplastic behavior of porous bronzes and irons," *Journal of Materials Processing Technology*, 113 (2001) 574-580.
6. B. Wang and D. Shu, "Experimental investigation on the viscoelastic properties of porous metals," *Journal of Materials Processing Technology*, 125-126 (2002) 144-149. N.E.Dowling, Mechanical Behavior of Materials, Prentice-Hall, Englewood Cliffs, NJ, 1993.
7. N.E. Dowling, *Mechanical Behavior of Materials*, Prentice-Hall, Englewood Cliffs, NJ, 1993