Viscoelastic behaviour of porous sintered steels compact

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This paper describes the viscoelastic behaviour of sintered steels with porosities of 12, 20 and 33%, using a dynamic mechanical analyser. Test specimens were prepared from premix powders of 100–150 μm size by a process of die compaction, delubrication and sintering. The influences of test temperature and vibrational frequency on storage and loss modulus and tangent delta have been investigated. The investigated operating temperature and frequency was varied from 25 to 280°C and from 10 to 50 Hz respectively.

Keywords: Steel, Viscoelastic behaviour, Temperature, Frequency effects

Introduction

The feasibility for the production of porous metallic components from powder metal by employing the powder metallurgy (PM) route of mixing, die compaction, delubrication and sintering is well recognised. Utilising this technology, porous materials are being prepared with known porosity and are being extensively used in the field of filtration, flame arresters, silencers, structures for gas dispersion in liquids, components for drying processes, air bearings, oil lubricated bearings, burners, heat exchangers and filters.[1–11] However, there are challenges in fabricating such porous metal structures using PM techniques, particularly the product possessing the combination of several properties including high mechanical strength combined with ductility and good damping capacity. Some of the articles specifically describing the damping capacity of porous materials revealed that the damping capacity of porous materials is significantly greater in the order of several times than that of parent materials.[12–15] Porous components manufactured from high density metal powder are always offer great interest owing to its ability to absorb high energy (high damping capacity) and to provide high strength rather than light weight components when heavy impact load fall on this material.[16–18] Studies have been performed to improve the strength of the porous powder metal components under dynamic loading.[5,19–21] In general, research on viscoelastic behaviour of polymeric materials and their composites are well advanced when compared to PM components, even though their applications are extensive. In the present study, the viscoelastic behaviour of sintered steel was investigated at porosities of 12, 20 and 33%, using a dynamic mechanical analyser (DMA).

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Experimental

Powder characteristics

A premixed steel powder (North America Hoganas Inc.) was used in this study with a composition of 2·06 wt-%Ni, 0·83 wt-%Mo, 0·26 wt-% graphite, 0·73 wt-% wax as lubricant and balanced Fe. Before compaction, the theoretical density of the powder was measured using a helium pycnometer at 7·64 g cm^{-3}. The powder morphology is irregular and most of the particles fall in the size range of 100–150 μm, as shown in Fig. 1.

Sample preparation

The powder was compacted with three different pressures of 55, 276 and 497 MPa into bar specimens of $37\times6\times4$ mm. The number of specimen prepared for each compaction pressure was three. The specimens were heated to 400°C at a heating rate of 5°C min^{-1} in a mixed atmosphere of 80%N_2 and 20%H_2 and allowed to dwell for 3 h to burn out all the lubricant. Sintering was carried out in the same atmosphere at a heating rate of 3°C min^{-1} to a peak temperature of 800°C for 1 h. The sintered density was calculated from its mass and dimensions, as shown in Table 1. Figure 2 shows the microstructure for porosities of 33 and 12%.

Dynamic mechanical analyser

The bending mode was used to evaluate the storage and loss modulus and tangent delta as a function of frequency and temperature. Before the test run in air, temperature, compliance, clamp and force calibration were performed according to the vendor recommended procedure DMA (model Q800, TA instruments DMA). The applied force set was 700 mN and the temperature varied from 25 to 280°C. The test frequency range was from 10 to 50 Hz. The stress field was within the elastic region for all of the samples.

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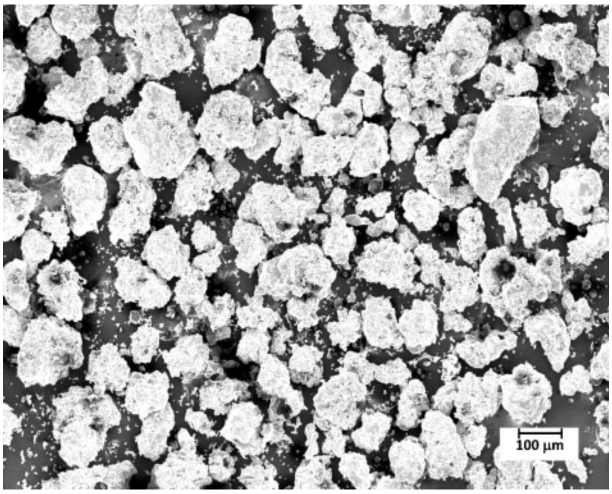
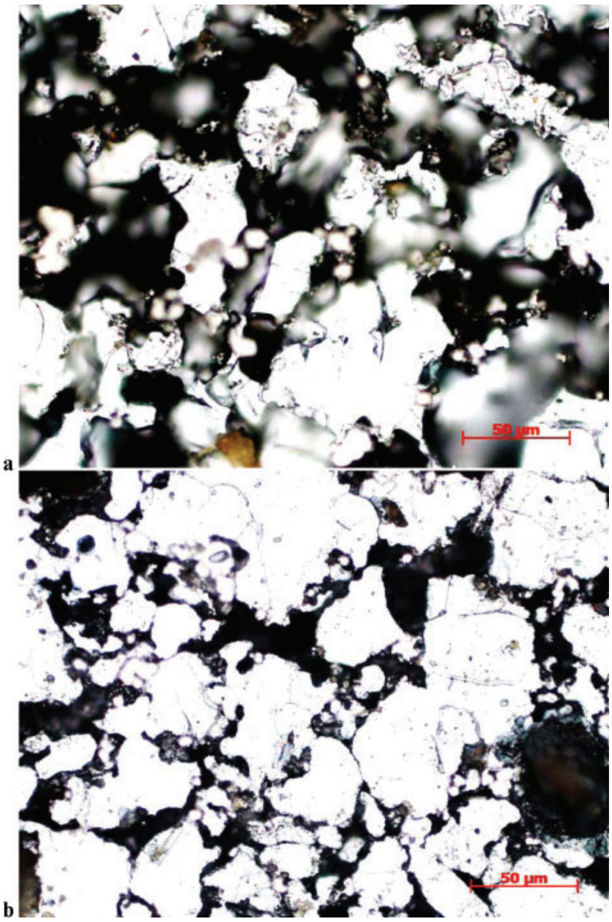


1 Scanning electron micrograph of premixed steel powder



a 33% porosity; *b* 12% porosity
2 Optical micrographs of sintered samples

Table 1 Properties of compacts

Compaction pressure, MPa	Sintered density, g cm^{-3}	Porosity θ, %
55	4·62	33
276	6·29	20
497	6·83	12

Results and discussion

Theoretical background of storage and loss moduli and tangent delta

Viscoelasticity indicates a material with the features of both a viscous fluid and an elastic solid. The term elastic and viscous nature is the represented action of spring and the response of putty system. In the first case, the return of spring to its original position when stretched and then released is an example of elastic action. In the second case, the retaining capability of the extended state when it is pulled is an example of viscous nature. Materials with a combination of these properties are called viscoelastic. The degree to which a metallic material behaves either viscously or elastically depends mainly on temperature and rate of loading (frequency). A generalised equation relates the viscoelastic properties of storage modulus E' and loss modulus E'' with complex modulus E^* as follows[22]

$$E^* = E' + iE'' \tag{1}$$

where i is the square root of -1. The absolute value of complex modulus E^* is Young's modulus E. Another viscoelastic parameter calculated in this study was tangent delta or $\tan\delta$, which is the ratio of E'' to E'. The definition of δ is the phase angle between the dynamic stress and the dynamic strain. An input sinusoidal stress σ can be expressed in time domain t with frequency ω and amplitude σ_a as follows

$$\sigma = \sigma_a \sin \omega t \tag{2}$$

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

$$\varepsilon = \varepsilon_a \sin(\omega t - \delta) \tag{3}$$

From equations (1)–(3), the following relations are obtained

$$|E^*| = E = \frac{\sigma_a}{\varepsilon_a} \quad E' = E\cos\delta \quad \text{and} \quad E'' = E\sin\delta \tag{4}$$

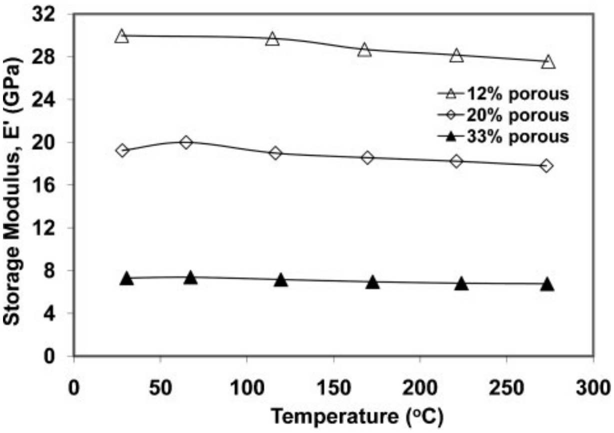
The final viscoelastic parameter used in this study is total energy dissipation Δu in each cycle per unit volume of material which can be expressed as follows

$$\Delta u = \sigma_a \varepsilon_a \sin\delta \tag{5}$$

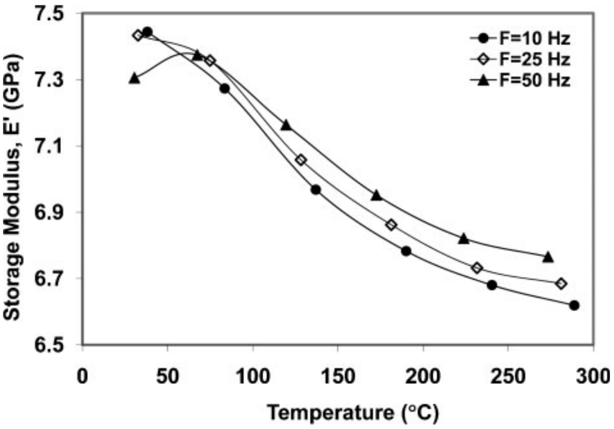
Storage modulus

Storage modulus E' is one of the important viscoelastic parameters used to measure the stiffness, representing the elastic portion. This parameter is associated with stored energy within a sample during a cycle. Elastic energy stored in the sample during loading is returned when the load is removed. As a result, the elastic stress and strain curves are completely in phase. For the measured elastic portion, Hooke's Law applies, where the stress is proportional to the strain and the modulus is defined by the ratio.

To investigate the effect of porosity θ with temperature T, the storage modulus E' was measured with porosities of 12, 20 and 33%. The operating frequency of the cyclic load was kept constant as 50 Hz and the temperature was ramped from 25 to 280°C at a heating rate of 5°C min^{-1}. Figure 3 plots the change in storage modulus against temperature for different porosities.

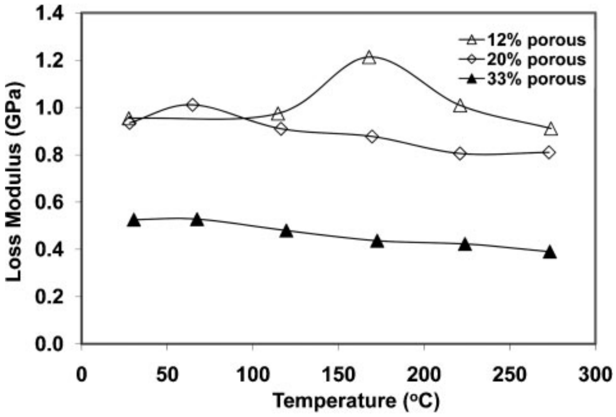


3 Storage modulus of sintered steel with 12, 20 and 33% porosity



4 Storage modulus of sintered steel *v.* temperature with different operating frequencies

The linear sensitivity to storage modulus was calculated with respect to porosity and temperature as shown in Table 2. The negative sign indicates a decrement of storage modulus as the porosity or temperature is increased. The decrease in storage modulus with 1% increment of porosity was 1·06 GPa at 25°C, 1·02 GPa at 150°C and 0·97 GPa at 280°C. Similarly, the decrease in storage modulus with 1°C increment of temperature was 1·04 GPa at 12% porosity, 0·75 GPa at 20% porosity and 0·27 GPa at 33% porosity. From these results, it can be concluded that the storage modulus



5 Loss modulus of sintered steel with 12, 20 and 33% porosity

shows a similar sensitivity to porosity in % and temperature in °C. The sensitivity to storage modulus was decreased as both porosity and temperature is increased.

To investigate the effect of frequency ω with temperature T, the storage modulus with frequencies of 10, 25 and 50 Hz was compared for a porosity of 33%. The operating temperature was ramped from 25 to 280°C at a heating rate of 5°C min^{-1}. The frequency dependence of storage modulus with increase in temperature is plotted in Fig. 4. The storage modulus decreases with a higher temperature except for the region below 50°C. The linear sensitivity to storage modulus was calculated with respect to frequency as given in Table 3. The increase in storage modulus with 1 Hz increment of frequency was $1{\cdot}25\times10^{-3}$ GPa at 150°C and $2{\cdot}56\times10^{-3}$ GPa at 280°C. On comparison with the effect of porosity in % and temperature in °C, the effect of frequency in Hz is smaller than other two effects.

Loss modulus

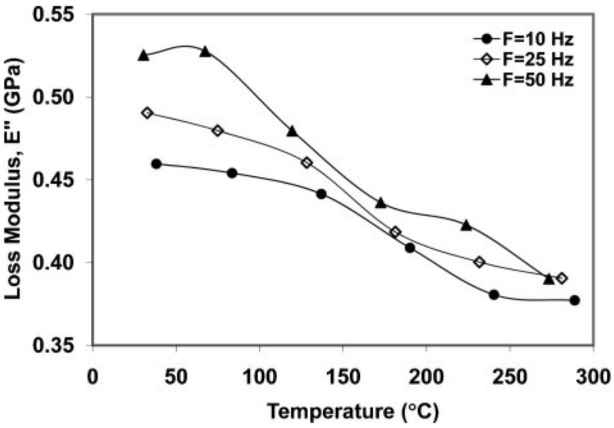
The loss modulus E'' representing the viscous portion is associated with the ability of materials to dissipate energy. The dissipated energy usually changes into heat. The effect of porosity θ with temperature T was investigated and the loss modulus with porosity compared while the operating frequency of the cyclic load was kept constant as 50 Hz but the temperature

Table 2 Linear sensitivity to storage modulus

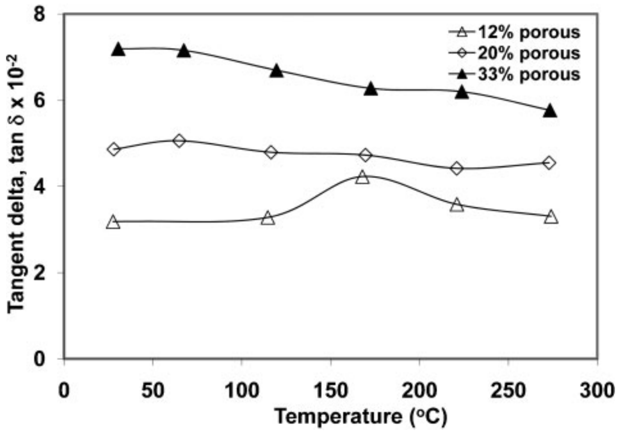
sensitivity at frequency of 50 Hz				sensitivity at porosity of 33 %	
$\partial E'/\partial\theta$ (GPa/%)	Temperature T (°C)	$\partial E'/\partial T$ (GPa/°C)	porosity θ (%)	$\partial E'/\partial\omega$ (GPa/Hz)	temperature T (°C)
–1·06	25	–1·04	12	–	25
–1·02	150	–0·75	20	$1{\cdot}25{\cdot}10^{-3}$	150
–0·97	280	–0·27	33	$2{\cdot}56{\cdot}10^{-3}$	280

Table 3 Linear sensitivity to loss modulus

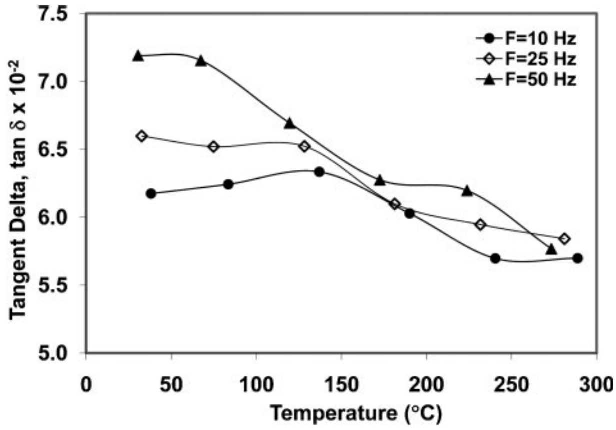
sensitivity at frequency of 50 Hz				sensitivity at porosity of 33 %	
$\partial E''/\partial\theta$ (GPa/%)	Temperature T (°C)	$\partial E''/\partial T$ (GPa/°C)	porosity θ (%)	$\partial E''/\partial\omega$ (GPa/Hz)	temperature T (°C)
$-3{\cdot}42{\cdot}10^{-2}$	25	–	12	–	25
$-3{\cdot}27{\cdot}10^{-2}$	150	$-7{\cdot}45{\cdot}10^{-4}$	20	$9{\cdot}89{\cdot}10^{-4}$	150
$-3{\cdot}13{\cdot}10^{-2}$	280	$-5{\cdot}99{\cdot}10^{-4}$	33	$1{\cdot}03{\cdot}10^{-4}$	280



6 Loss modulus of sintered steel *v.* temperature with different operating frequencies



7 Tangent delta of sintered steel with 12, 20, and 33% porosity



8 Tangent delta of sintered steel *v.* temperature with different operating frequencies

was ramped from 25 to 280°C at a heating rate of 5°C min^{-1}.

The dependence of loss modulus on porosity, temperature and frequency is shown in Figs. 5 and 6. The loss modulus is much smaller than the storage modulus for all samples, which means the porous Fe alloy samples exhibit mostly elastic behaviour. The 12% porosity sample does not show the same trend as the other two samples; however, it appears below the test sensitivity range does not have enough viscous behaviour for the DMA test. Therefore, the linear sensitivity to loss modulus was calculated with respect to porosity (only to 20 and 33%, not to 12%) and temperature as listed in Table 2. From these results, it can be concluded that the loss modulus shows higher sensitivity to porosity in % than to temperature in °C.

To investigate the effect of frequency ω with temperature T, the loss modulus with frequencies of 10, 25 and 50 Hz was compared for a particular porosity of 33% while the operating temperature was ramped from 25 to 280°C at a heating rate of 5°C min^{-1}. The linear sensitivity to loss modulus was calculated with respect to frequency as listed in Table 2. On comparison with the effect of porosity in % and temperature in °C, the effect of frequency in Hz is smaller than the porosity effect but similar to temperature on changing the loss modulus. Note that the loss modulus behaviour loses linearity and becomes non-linear as the frequency increases.

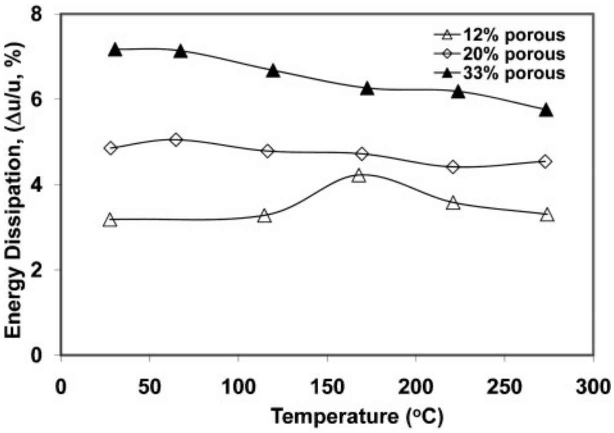
Tangent delta

Tangent delta is a measure of the ability to dissipate strain energy during mechanical vibration under cyclic loading.[23] When a structure is subjected to oscillatory deformation, it acquires both kinetic and potential energy and there is energy dissipation on each cycle. The amount of energy dissipation is experimentally quantified in terms of damping level. In metallic materials, this behaviour is closely associated with the micro- and macropores, grain boundaries and dislocations.[24] The damping capacity of porous materials is far greater than that of the non-porous materials. The damping capacity of porous materials increased with porosity at a rate several times higher than that of the parent materials.[12–15] In general, a high value of tangent delta indicates that the structure has a high damping capacity; a low value represents high elasticity and low damping capacity. The effect of porosity θ with temperature T was investigated and the tangent delta with porosities of 12, 20 and 33% compared while the operating frequency of the cyclic load was kept constant as 50 Hz but the temperature was ramped from 25 to 280°C at a heating rate of 5°C min^{-1}.

The damping measurement is shown in Figs. 7 and 8. In this work, the phase angle was determined at the start of the linear portion of the storage modulus–dynamic force curve at a fixed dynamic force of 700 mN. The

Table 4 Linear sensitivity to tangent delta

	Sensitivity at frequency of 50 Hz			Sensitivity at porosity of 33%
Temperature T, °C	$\partial\tan\delta/\partial\theta$, %/%	$\partial\tan\delta/\partial T$, %/°C	Porosity θ, %	$\partial\tan\delta/\partial\omega$, %/Hz
25	0·177	–	12	–
150	0·140	$-5{\cdot}66\times10^{-3}$	20	$1{\cdot}12\times10^{-2}$
280	0·100	$-9{\cdot}60\times10^{-3}$	33	–



9 Energy dissipation of sintered steel with 12, 20 and 33% porosity

linear sensitivity to loss modulus was calculated with respect to porosity and temperature as listed in Table 4. From these results, it can be concluded that the loss modulus shows higher sensitivity to porosity in % than to temperature in °C. Note that the samples of two higher porosities were only investigated.

To investigate the effect of frequency ω with temperature T, the tangent delta with frequencies of 10, 25 and 50 Hz was compared for a particular porosity of 33% while the operating temperature was ramped from 25 to 280°C at a heating rate of 5°C min^{-1}. The linear sensitivity to loss modulus was calculated with respect to frequency as listed in Table 3. On comparison with the effect of porosity in % and temperature in °C, the effect of frequency in Hz is smaller than the porosity effect but similar to temperature on changing loss modulus. Note that the tangent delta behaviour with frequency was not linear so the linear sensitivity was calculated only at 150°C.

Energy dissipation

The total energy dissipation Δu was calculated in each cycle per unit volume of material from equation (5). The results are very similar to tangent delta, as shown in Fig. 9, because $\delta \approx \sin\delta \approx \tan\delta$ at small δ. As for the porous Fe alloy samples used in this study, 3–7% of total energy applied is dissipated every cycle.

Conclusions

There is a relation between the porosity, temperature and frequency on change in storage modulus, loss modulus, tangent delta and energy dissipation of the steel compacts. Within the range of experiment, the followings can be concluded:

(i) porosity decreases the storage and loss moduli and increases the tangent delta and energy dissipation
(ii) high temperature decreases the storage and loss moduli, and tangent delta, and energy dissipation
(iii) high vibrational frequency increases the storage and loss moduli, and tangent delta, and energy dissipation.

Furthermore, the viscoelastic parameter of porous sample follows the following order:

(i) storage modulus: porosity$\geqslant$ temperature$\gg$frequency
(ii) loss modulus: porosity$\gg$ temperature$\approx$frequency
(iii) tangent delta and energy dissipation: porosity$\gg$ frequency$>$temperature.

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