

Morphology and damping characteristics of woodceramics

ZHANG DI, XIE XIAN-QING, FAN TONG-XIANG

State Key Laboratory of Metal Matrix Composites, Shanghai Jiao Tong University, Shanghai, 200030, People's Republic of China

E-mail: xiexqsh@yahoo.com.cn

T. OKABE, T. HIROSE

Industrial Research Institute of Aomori Prefecture, Hirosaki, 036-8363, Japan

Woodceramics has attracted more and more attention as ecomaterial at low cost in recent years. The purpose of this paper is to investigate morphology, damping characteristics of woodceramics and relationship between them. The experimental results reveal that pores in woodceramics can be divided into macropores, micropores and nanopores according to their dimensional size. Macropores are the majority of interconnected pores while most micropores are closed pores. Based on formation process, nanopores are supposed to consist of isolated nanopores and dendritic nanopores. Woodceramics exhibits significant damping gains, especially at room temperature, higher strain amplitude and higher vibration frequency. The values of damping capacity increase with vibration frequency and strain amplitude, decrease with testing temperature. Macropores and some nanopores have little but micropores and some other nanopores have much contribution to damping properties of woodceramics. The possible damping mechanisms of woodceramics at normal temperature are defect damping and thermoelastic damping; at high temperatures, interface damping is likely to be responsible for the majority of the damping of woodceramics.

© 2002 Kluwer Academic Publishers

1. Introduction

With worldwide economy developing quickly, we are confronting how to deal with excess garbage, such as disused wood in construction industry, waste paper and telephone book in everyday life, sugarcane dregs and apple dregs in food industry and so on. At present the usual methods to deal with such garbage are burying and burning, which damage environment badly. If these waste materials can be reused effectively, it is beneficial for reducing resource using and environmental protection. Woodceramics, a new kind of porous ceramic material [1, 2], can be fabricated from above-mentioned waste materials that are impregnated with phenol resin using an ultrasonic impregnation system and then carbonized at a given temperature. Thus woodceramics is environment conscious materials designed for minimizing the environmental impacts [3]. This exceptionally lightweight material possesses a unique combination of special properties [4, 5], such as electromagnetic shielding properties, friction and wear properties and so on. Hence woodceramics is expected to improve the performance of many new products, which has been described in detail in elsewhere [1]. Thus woodceramics has drawn special attention from all over the world. Typical production routine of woodceramics from *Pinus radiata* is illustrated in Fig. 1.

For applications of many structural materials or some functional materials, a knowledge of the damping prop-

erties is of great importance. Generally, the damping capacity is a measure of the energy that is dissipated to the surrounding environment in the material during mechanical vibration under cyclic loading [6]. In most application, a high damping capacity is desired. When woodceramics is applied in electrical brush, brake disk, bearing bush or other, it is inevitable that mechanical vibration would be produced when they are in use, which would disturb the regular application of facility, even make them failing, so it is very necessary to investigate the damping property of woodceramics. Inspection of the relevant scientific literature shows that, damping capacity of woodceramics has not yet been reported. Moreover, for many applications of porous materials, surface morphology and pores character are often very important since they have much effect on their properties. For instance, R.W. Rice [7] reported that SiC fiber composites made by CVI have decreasing Young's modulus with porosity increasing, and pores in different shape contribute different amount to that. H. Priellip [8] found that toughness of porous Al_2O_3 decreases from $1.8 \text{ MPa} \cdot \text{m}^{1/2}$ to $1.5 \text{ MPa} \cdot \text{m}^{1/2}$ with porosity increasing from 25 vol% to 35 vol%. However, to our best knowledge, morphology character of woodceramics and its effect on damping properties have not been reported. In present work, morphology character of woodceramics is observed and analysed. Damping capacity of woodceramics under various testing

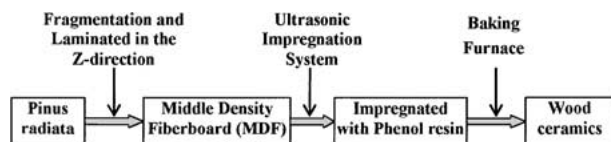


Figure 1 Schematic representation of the production of woodceramics from *Pinus radiata*.

conditions is investigated. The effect of morphology on damping behavior and damping mechanisms of woodceramics, at normal temperature and elevated temperature, are discussed respectively.

2. Experimental procedure

Woodceramics specimens were prepared from medium-density fiber board (MDF) made from *Pinus radiata* and impregnated with phenolic resin by sintering at 800°C, 1000°C and 1200°C in vacuum furnace (these samples were denoted hereafter with WCMs800, WCMs1000 and WCMs1200 respectively). The apparatus used here was described in detail in [1, 2]. The MDFs were impregnated with phenolic resin in a ratio of 1:1 by weight under decompressed condition prior to burning. Scanning electron microscopy (SEM) was employed to investigate the micrograph of woodceramics. The test-pieces with dimension of $40 \times 3 \times 1 \text{ mm}^3$ were machined by homemade clamp. The tests were carried out using a dynamic mechanical thermal analyzer (DMTA, type MK-IV) with three-point flexure mode. The test-pieces were constrained at two ends by clamping plates and excited into a sinusoidal flexural vibration by vibrator acting at its middle place. Fig. 2 showed the test-pieces preparation and loading orientation of woodceramics. Wood fibers were laminated in the Z-direction and the load was applied in Z-direction. In present work, material damping was characterized in terms of the suppression of resonant amplitude and the phase lag of deformation behind the applied load in forced vibration. The damping capacity was evaluated in a loss tangent ($\tan \phi$) using the following equation,

$$\tan \phi = E''/E'$$

where E' and E'' are the storage and loss moduli, respectively. E' corresponds to Young's modulus, while E'' represents the energy dissipated.

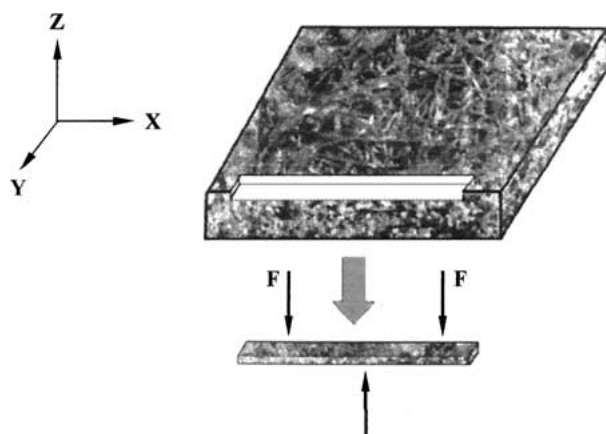


Figure 2 Specimens preparation and loading orientation of woodceramics for the present measurements.

3. Results and discussion

3.1. Micrograph characterization of woodceramics

Fig. 3 shows the horizontal section (X – Y plane in Fig. 2) morphology of WCMs1000 made from MDF. Clearly, carbonized wood fibers cross randomly on X – Y plane. There are three types of pores in woodceramics based on their dimensional size, namely macropore, micropore and nanopore. Fig. 4 shows corresponding higher-magnification image of Fig. 3. It is seen that the carbonized wood fibers are bridged with each other, and macropore come into being among the inter-fiber, and most of these macropores provide an interconnected continuous channel of woodceramics. The three-dimensional (3-D) network of interconnected macropores appears to have some anisotropy, and the pores structures are inhomogeneous. They have no regular shape and their sizes range from $10 \mu\text{m}$ to $100 \mu\text{m}$, which is decided with the diameter, orientation and impact degree of wood fiber. Obviously, normal lines of most of these macropores are roughly parallel to woodceramics laminated direction. From this figure, one also can see some single micropores (marked by A).

Fig. 5 presents the grouped micropores image in WCMs1000. In contrast to macropores, micropores have regular shape, and most of their cross section morphologies are rounded or elliptic, which is decided just with the type of wood fiber. Their dimensional sizes range from $1 \mu\text{m}$ to $10 \mu\text{m}$, which is decided just with sintering temperature of woodceramics, independent of fibers structural characteristics. We can see that the normal lines of most of these micropores are perpendicular to laminated direction. It should be noted here that most of the micropores are fibriform tracheids in wood fiber, thus they can't connect with each other due to the separation of fiber wall. In other words, they are closed pores.

Nanopores in woodceramics can't be observed by electrical microscopy due to the very small size, but they can be measured. This has been done by Fan using micromeritics tester (type ASAP2010) [9] and his results show that the average nanopores diameters range from 10 nm to 20 nm. These pores have different contribution when woodceramics is applied in different practical engineering. Macropores act as a most important role when woodceramics is used as structural material [10]. However, micropores by themselves contribute only a limited amount to decrease of mechanical properties because of its small quantity and tubular shape. However, when woodceramics is used as sorbent, nanopores play a key role due to they increasing specific surface area of woodceramics remarkably, while both macropores and micropores have little contribution to it.

Fig. 6 shows schematic diagram of the formation process of pores in woodceramics. Clearly, macropores and micropores just with smaller sizes already exist in woodceramics before sintering. With temperature increasing during sintering process, a series of reactions would take place in woody component and phenol resin, such as dehydration, fission, dehydrogenation, carbonization and so on (Fig. 7), and large molecules gradually depolymerize to small molecules. Some volatiles, such as CO, CO₂, vapor, CH₄ and so

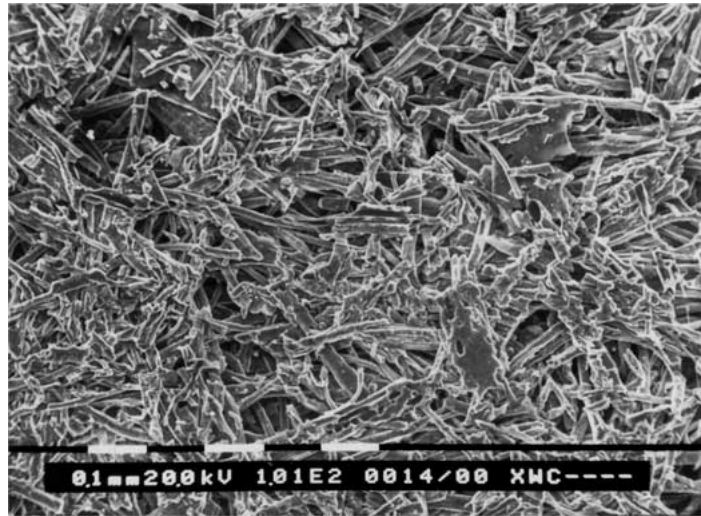


Figure 3 SEM image of horizontal section of WCMs1000.

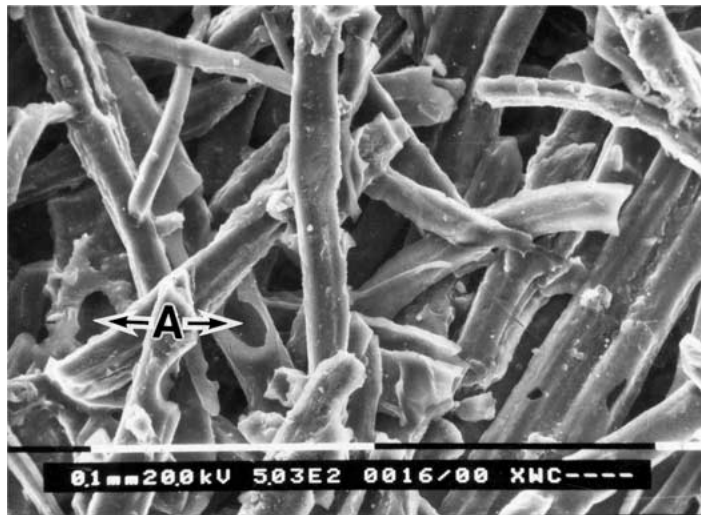


Figure 4 Macropores in WCMs1000.

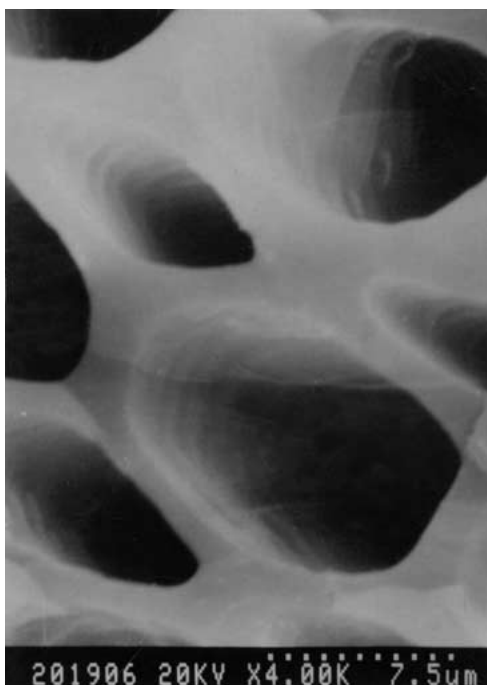


Figure 5 Micropores in WCMs1000.

on, are generated and most of them are removed. As a result, the volumes of wood fibers shrink, and macropores and micropores become larger with sintering temperature increasing. At the same time, the escape process of decomposition gases and partial loosening in wood fiber due to aforementioned degradation reactions probably together result in the formation and grown-up of nanopores. Initially, decomposed gases disperse in cell wall, forming “gas point”. Due to very low gas pressure, some gas points that can’t move become isolated nanopores (described in Fig. 7 with dot). Besides, there also exist some movable gas points simultaneously to form dendritic nanopores (described in Fig. 7 with branch-like symbol). When gas points move in cell wall of wood fiber, surface energies and partial loosening of wood fiber are likely to result in such gas points gradually merging to form larger ones, as a result, pressure in gas point increases, which is beneficial to gas points escaping out of wood fiber. A detailed illustration for dendritic nanopores is presented in Fig. 8. It should be noted here that the quantities of nanopores in woodceramics in Fig. 7 don’t reflect the true situation. In fact, the density of nanopores is very high.

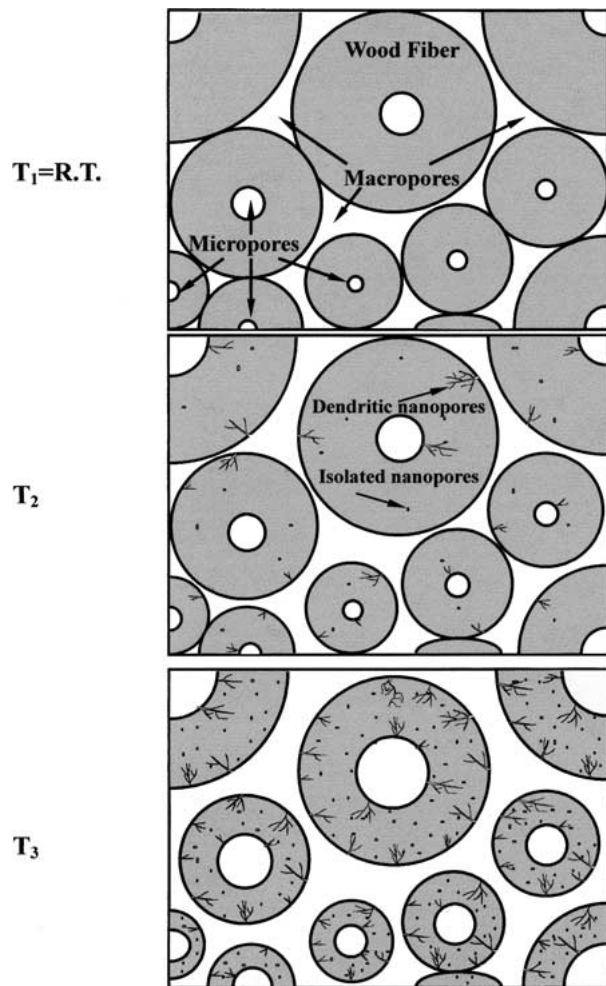


Figure 6 Schematic diagram of the formation process of pores in wood-ceramics ($T_1 < T_2 < T_3$).

With higher sintering temperature in baking furnace, aforementioned reactions would proceed more thoroughly. Consequently, partial loosening degree in wood fibers increases, which favors gas point escaping. At the same time, the quantities of gas points also increase

Wood Fiber Outer Surface

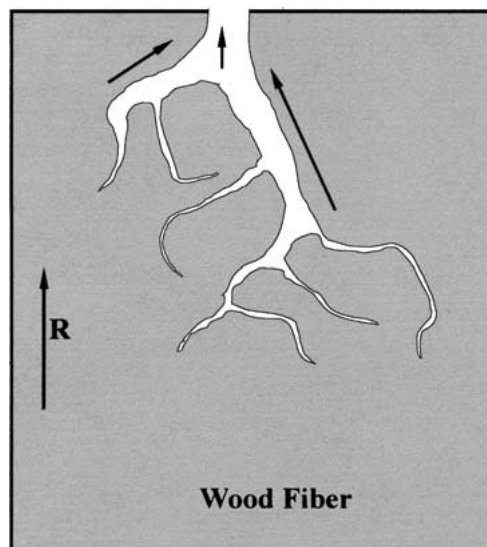


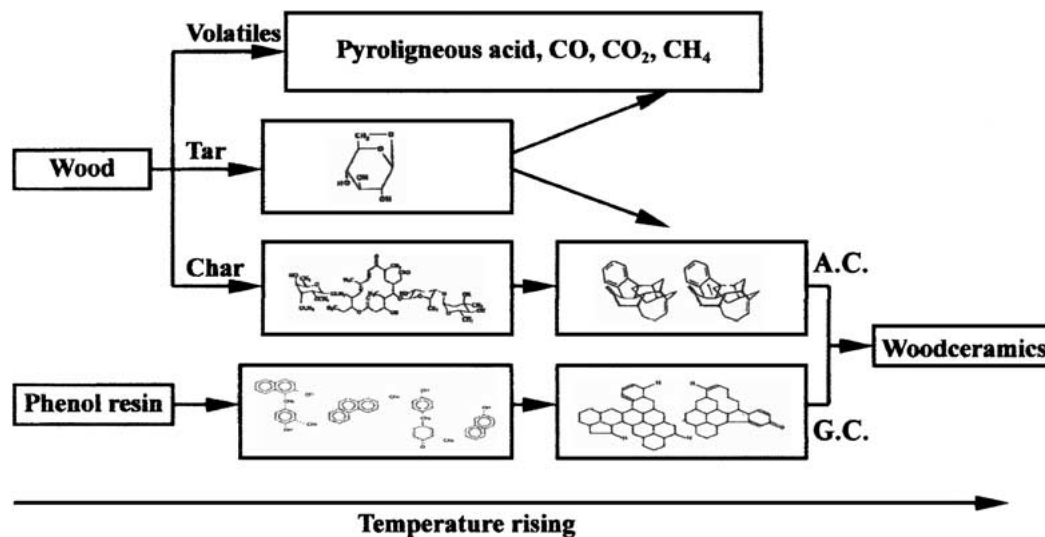
Figure 8 Detailed illustration for dendritic nanopore.

with sintering temperature, which increases the probability of merging. Moreover, according to Clapayron equation, gas pressure increases with sintering temperature, which makes it easier for gas point to escape out of wood fibers. Thus, the combination of these factors probably may lead to the result that the quantity of isolated nanopores increases slowly and that of dendritic nanopores increases rapidly with sintering temperature increasing. Hence the total quantity of nanopores increases with sintering temperature increasing.

3.2. Damping capacity

The characterization of the damping capacity of wood-ceramics is very sensitive to testing conditions, such as testing temperature, strain amplitude, frequency and so on. They will be discussed below in detail.

Many materials have frequency dependent damping [11]. Metal matrix composites, including Al alloy



A.C. denotes amorphous carbon, G.C. denotes glassy carbon.

Figure 7 Thermal decomposition products of woodceramics. A.C. denotes amorphous carbon, G.C. denotes glassy carbon.

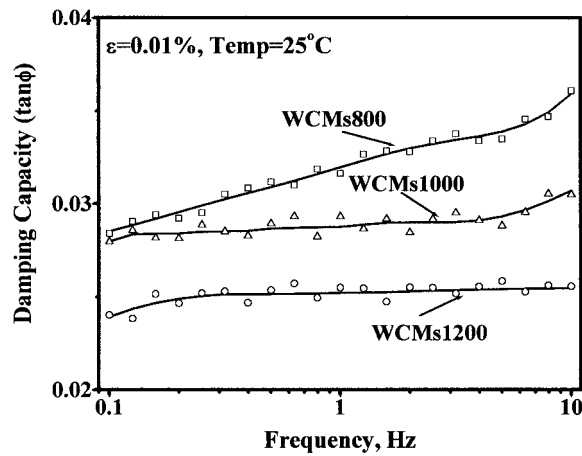


Figure 9 Frequency dependence of damping capacity for Woodceramics.

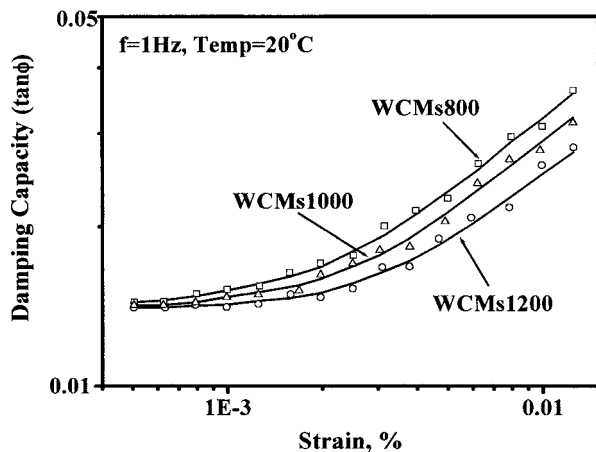


Figure 10 Strain dependence of damping capacity for woodceramics.

matrix and Mg alloy matrix, show a decreasing damping with increasing frequency, and decrease rate at room temperature is smaller than that at elevated temperature. However, for porous materials, to the best of our knowledge, there is no work reported in the literature concerning the influence of frequency on damping capacity. The damping capacity woodceramics of woodceramics as a function of frequency is presented in Fig. 9. Clearly, with frequency increasing, damping capacity of woodceramics shows inverse variation of that of composites. At room temperature, damping capacity values of all the woodceramics increase with frequency increasing within entire frequency range, and that of woodceramics sintered at lower temperature has higher increase rate.

Fig. 10 shows the damping capacity vs. strain amplitude of the woodceramics. From this figure, it is clear that damping capacity values of woodceramics increase with increasing strain amplitude at room temperature, and their increase rates are controlled by woodceramics type and strain amplitude level. Woodceramics sintered at higher temperature has lower increase rate. For each type of woodceramics, at higher strain amplitude, the values of damping capacity increase more rapidly than those at lower strain amplitude do. This is consistent with the behavior expected in other materials. For silicon carbide with porosity over 14% as an example, the damping increased smoothly with the strain amplitude at room temperature [12]. 6061Al/Gr composites

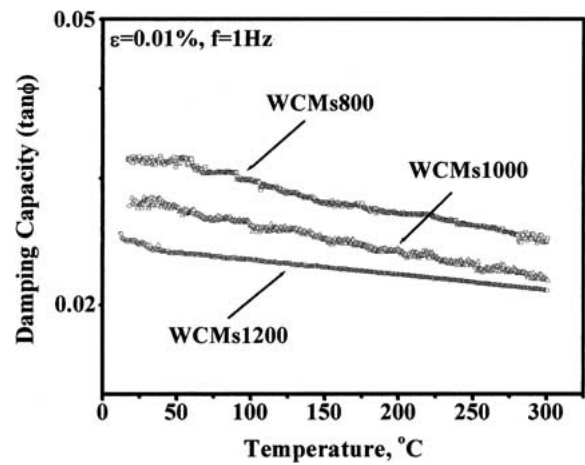


Figure 11 Temperature dependence of damping capacity for woodceramics.

also show a increasing damping with strain amplitude increasing [13].

The testing temperature response of the damping capacity for the woodceramics is depicted in Fig. 11. The damping capacity values of all the samples decrease with increasing testing temperature, and woodceramics sintered at higher temperature has smaller decrease rate. This phenomenon isn't in agreement with the reported observations on most other material, whose damping capacity values increase with testing temperature increasing.

3.3. Damping mechanisms of woodceramics

3.3.1. Damping mechanism at normal temperature and effect of pores on damping behavior of woodceramics

The damping mechanisms for most materials mainly include the following three types [14], magnetic damping, thermoelastic damping and defect damping. The former two types generally result from the bulk response of a material [15, 16]. Two possible damping mechanisms of woodceramics are supposed to be thermoelastic damping and defect damping.

Thermoelastic damping may occur when the material undergoes a heterogeneous deformation [16]. During DMTA testing in present investigation, test pieces experience three-point bending, thus thermoelastic damping may take place and then can be characterized by losses incurred in the irreversible flow of heat from the compression side to the cooler tension side, but compared with other damping mechanisms, thermoelastic damping often is small, just with the order of 10^{-5} or so [14].

Defect damping always occurs on common vibration condition. If defects move nonreversibly when sample receives external circular load, the energy would be dissipated and defect damping occurs. For woodceramics, aforementioned defects may include point defect (vacancies and interstitial atoms), planar defect (phase interface) and bulk defect (porosity).

Both amorphous carbon and glass carbon in woodceramics belong to noncrystalline carbon, in which carbon atoms array disorderly. Consequently, plenty of vacancies and interstitial atoms may exist in or between

layers of carbon atom. With external load, these vacancies and interstitials may move nonreversibly and the system energy may be dissipated.

During the fabrication process of woodceramics, amorphous carbon from wood component and glassy carbon from phenol resin come into being. The properties of amorphous carbon, such as mechanical properties, physical properties, chemical properties and so on, are different from those of glassy carbon, thereby woodceramics is actually a carbon/carbon composite, and there must exist a large interface between glassy carbon and amorphous carbon. As a result, interface would contribute much to damping of woodceramics.

As for the pores effect on damping behavior in present investigation, there are two circumstances. It is well known that porosity enhances damping due to stress concentration and mode conversion around pores, and an external force can induce atomic rearrangements and viscous-like sliding of atoms relative to each other [17]. During the fabrication process of woodceramics, phenol resin, changing into glassy carbon, covers wood fibers, which change into amorphous carbon. Thus distribution of glassy carbon in amorphous carbon isn't homogeneous. As is known, glass carbon is a kind of carbon material that possesses some special properties, such as high strength, high modulus, high chemical stability, low density and so on [18]. In deep cell wall of woodceramics, there is less glassy carbon in the region, thus Young's modulus is supposed to be lower. As a consequence, stress concentration and mode conversion may occur easily around the pores (basically including micropores and some nanopores) in this region, and there will be an impedance mismatch to vibration movement between the gas in the pores and the cell wall of woodceramics. This mismatch may change the deformation field in the neighbouring region and therefore lead to secondary shear deformation in cell wall around the pores, and thereby increasing the damping capacity. On the other hand, in the region near outer surface, there is more glassy carbon. Due to strengthening effect of glassy carbon, stress concentration and mode conversion are supposed to be difficult to generate around these pores (mainly including macropores and some other nanopores) in woodceramics, thus pores in this region have less contribution to damping capacity.

3.3.2. Damping mechanism at elevated temperature

The factors influencing elevated-temperature damping properties of woodceramics mainly include intrinsic damping of amorphous carbon and glassy carbon and interface damping between them. Within testing temperature range, intrinsic damping of amorphous carbon and glassy carbon change little [19], thus they have little contribution to the change of elevated-temperature damping of woodceramics, thus interface damping is supposed to be the main factor influencing elevated-temperature damping properties of woodceramics.

The effect of perfectly bonded interfaces on damping may be rationalized on the basis of the Schoeck theory [20]. In the theory, damping capacity is increased by interface relaxation and anelastic strain induced by

dislocations in the vicinity of the interface. The effect of weakly bonded interfaces on the overall damping capacity has been analysed using interface slip model [21]. In the model, interface damping is attributed to the frictional energy loss at the interface under cyclic loading. These two mechanisms often exist simultaneously, thus interface damping involves two processes, frictional damping and anelastic damping:

$$Q^{-1} = Q_1^{-1} + Q_2^{-1} \quad (2)$$

where Q^{-1} is interface damping, Q_1^{-1} and Q_2^{-1} are frictional damping and anelastic damping respectively. As mentioned earlier, since woodceramics have very large area of interface, frictional damping in woodceramics is much larger than anelastic damping. Hence interface damping of woodceramics depends on interfacial slip.

4. Conclusion

Several important conclusions can be drawn from this work:

1. Pores in woodceramics are divided into macropores, micropores and nanopores according their size. Based on formation process, nanopores are supposed to consist of isolated nanopores and dendritic nanopores. Macropores are the majority of interconnected pores while most micropores are closed pores. With sintering temperature increasing, macropores and micropores become larger, and the quantity of nanopores also increases.

2. Woodceramics exhibits significant damping gains, especially under the conditions of room temperature, high strain amplitude and high frequency. The values of damping capacity of woodceramics decrease with testing temperature, and increases with strain amplitude and vibration frequency.

3. Macropores and some nanopores have little contribution to woodceramics damping behavior and micropores and some other nanopores have much effect on damping properties of woodceramics. This is supposed to be related with different strengthening effect of glassy carbon.

4. The damping mechanisms of woodceramics may be ascribed to defect damping and thermoelastic damping. Defect damping, including point defect damping, interface damping and micropores (microcracks) damping, is supposed to be the main contributor. At elevated temperature, interface damping seems to play a dominant role in the damping of woodceramics.

Acknowledgements

The authors wish to acknowledge the financial support of the Research Fund for the Doctoral Program of Higher Education (No. 2000024823), Research fund of Science and Technology Commission of Shanghai Municipality (No. 00XD14017) and Fund for Cross-century Talents of the Ministry of Education, P.R. China.

References

1. T. OKABE, K. SAITO and K. HOKKIRIGAWA, *J. Porous Materials* 2 (1996) 207.

2. T. OKABE, K. SAITO, M. FUSHITANI and M. OTSUKA, *ibid.* **2** (1996) 223.
3. K. HOKKIRIGAWA, T. OKABE and K. SAITO, *J. Soc. Mat. Sci., Japan* **44**(501) (1995) 794.
4. T. AKAGAKI, K. HOKKIRIGAWA, T. OKABE and K. SAITO, *J. Porous Materials* **6** (1999) 197.
5. T. OKABE, K. SAITO, H. TOGAWA and Y. KUMAGAI, *J. Soc. Mat Sci., Japan* **44**(498) (1995) 288.
6. J. ZHANG, R. J. PEREZ, C. R. WONG and E. J. LAVERNIA, *Mat. Sci. Eng.* **R13** (1994) 325.
7. R. W. RICE and D. LEWIS III, in "Reference Book for Composites Technology-1," edited by S. M. Lee (Technomic Press, Lancaster, PA, 1989) p. 117.
8. H. PRIELIPP, M. KNECHTEL, M. CLAUSSEN, S. K. STREIFFER, H. MULLEJANS, M. RUHLE and J. RODEL, *Mat. Sci. Eng. A* **197** (1995) 19.
9. T. X. FAN, T. HIROSE, T. OKABE and D. ZHANG, *J. Porous Mat.* **8** (2001) 211.
10. H. IIZUKA, M. FUSHITANI, T. OKABE and K. SAITO, *ibid.* **6** (1999) 175.
11. E. J. LAVERNIA, R. J. PEREZ and J. ZHANG, *Metal. & Mat. Trans.* **26A** (1995) 2803.
12. A. WOLFENDEN, C. B. PROFFITT and M. SINGH, *J. Mat. Eng. and Perform* **8** (1999) 598.
13. R. J. PEREZ, J. ZHANG and M. N. GUNGOR, *Metall. Trans.* **24A** (1993) 701.
14. E. J. LAVERNIA, J. ZHANG and R. J. PEREZ, *Key Eng. Mat.* **104-107** (1995) 691.
15. C. ZENER, "Elasticity and Anelasticity of Metals" (University of Chicago Press, Chicago, IL, 1948) p. 147.
16. V. K. KINRA and K. B. MILLIGAN, in "M³D, Mechanics and Mechanisms of Material Damping," edited by V. K. Kinra and A. Wolfenden (ASTM, Philadelphia, PA, 1992) p. 94.
17. J. BANHART, J. BAUMEISTER and M. WEBER, *Mat. Sci. Eng. A* **205** (1996) 221.
18. B. MEHROTRA, R. H. BRAGG and A. S. RAO, *J. Mater. Sci.* **18** (1983) 2671.
19. X. N. ZHANG, Ph.D. Dissertation, Shanghai Jiao Tong University, 1997, p. 32.
20. G. SCHOECK and E. BISOGNI, *Phys. Stat. Sol.* (1969) 32.
21. N. N. KISHORE, A. GHOSH and B. D. AGARWAL, *J. Rein. Plast. Comp* **1** (1982) 41.

*Received 21 August 2001
and accepted 20 June 2002*