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Studies into the Physical Properties of a Ceramic Material with Deposited Film of Lubricant

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Abstract: Results of a study, using internal friction method, into elastic and damping properties of ceramics covered with a thin film of various lubricants are presented. It was found that both elastic modulus and damping coefficient are affected by the film of lubricant deposited on the surface of ceramic material $Al_2O_3*ZrO_2$. Extreme pressure type of additive to the lubricant proved to be especially effective in reducing modulus of elasticity of the ceramic studied.

1 INTRODUCTION

Studies, using internal friction method, into such physical properties of solids as their elastic and inelastic behaviour are well known. The internal friction method consists of measuring the phase difference between stress and strain during application of an alternating load to a sample. In particular, this method allows for an accurate characterisation of a number of relaxation processes within a sample taking place at a specific frequency of load application or temperature level and related to significant losses of vibration energy. These losses are clearly seen when the sample is properly isolated from its surroundings.

It has been found[1,2] that the presence of a thin layer of an organic fluid on the surface of metal sample considerably affects its vibration characteristic and modulus of elasticity. These effects are particularly strong near the phase transition temperatures of the material. Complete explanation of this phenomenon requires thorough investigation of the structure and nature of the fluid/solid interface and reliable characterisation of the substrate.

In this paper, results of studies into the elastic and damping properties of a ceramic material ($Al_2O_3*ZrO_2$) covered by a thin layer of a lithium-based grease are presented. The results are compared with those obtained for ferrous material under nominally the same experimental conditions.

2 EXPERIMENTAL MATERIALS AND PROCEDURE

A simple test apparatus[3] based on the principle of a torsional pendulum was used to measure the damping and elastic properties of the material studied. Frequency of measured vibrations was around 40 Hz. Internal friction, Q^{-1}, being a measure of vibration damping, was determined from the logarithmic decrement of damping, δ, as:

$$Q^{-1} = \frac{\delta}{\pi}$$
$$= n^{-1} \ln(A_1/A_{n+1})$$

where A_1 and A_{n+1} represent values of the first and $(n + 1)$ amplitude.

Frequency, f, of vibration for a given modulus of elasticity G was simultaneously measured with damping. Values of elasticity modulus given by the relation $f^2 \approx G$ are presented in arbitrary units because of difficulties in preparation of small ceramic samples with closely defined shape. Frequency was measured with high precision and the error of measurement does not exceed 0·1%. Both measured quantities, that is Q^{-1} and G, were determined in a continuous way as a function of temperature ranging from 140 to 670 K. The rate of temperature increase was 1°/min. Amplitude of deformation was of the order of 7×10^{-6} m.

Ceramic samples used were in the form of rods 32 mm long with a square cross-section with the side of 2 mm. Samples were fixed in the holder both mechanically and using special adhesive. Before every experiment, the sample was carefully chemically cleaned and kept for 30 min in vacuum (10^{-3} torr) at a temperature of 670 K. The internal friction spectrum for a cleaned ceramic sample was not affected by the cleaning process. Next, the sample was covered with a grease film. In order to secure maximum precision and repeatability of measurements, the sample, after cleaning, remained attached to the pendulum during the entire experiment. Prior to grease film deposition, vibrational damping of the specimen without surface film was determined. Afterwards, a film of grease was deposited in the following way. A small amount of grease, heated up to 340 K to reduce its viscosity, was spread over the specimen surface with the help of a glass rod. Next, the sample with deposited film of grease was heated up to 330–340 K in the test apparatus under vacuum of 10^{-2} torr. An even distribution of grease molecules over the surface could thus be achieved. The specimen with deposited film of grease was then cooled down to 150 K and was regarded as being ready for vibrational damping measurements. The thickness of the grease film was estimated to be around 50 μm for ceramic and 15 μm for steel. This estimate is based on the increase in mass of the specimen resulting from deposition of the grease film. It must be added, however, that, because of the porosity, the real thickness of the film could be considerably less than the above estimate.

It was found that the integrity of the grease produced by this method was not affected by the test conditions, i.e. temperature of 370–380 K and vacuum of 10^{-2} torr. In order to ensure proper contact between grease film and the substrate, the sample, when in the test apparatus, was heated up to a temperature of 330 K. After slow cooling of the system to 140 K, the measurements of damping and modulus of elasticity were carried out in vacuum of 10^{-2} torr while the sample was gradually heated up. In order to prevent vaporisation of thin grease film it was necessary to maintain vacuum conditions during measurements.

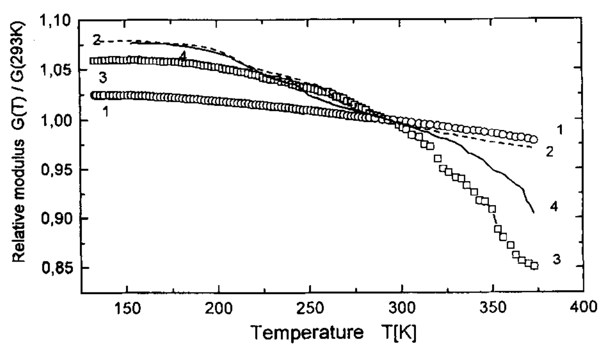

Fig. 1. Relative modulus as a function of temperature. (1) Mild steel without film of lubricant; (2) mild steel with deposited film of lithium-based grease containing 3% of graphite additive; (3) ceramic without film of lubricant; (4) ceramic with deposited film of lithium-based grease containing 3% of graphite additive.

The majority of tests were carried out on ceramic material $Al_2O_3{*}ZrO_2$ covered with straight lithium-based grease. In addition, the following compounds were added to the grease: 3% by weight of graphite; 3% by weight of molybdenum disulphide; 2% by weight of extreme pressure agent, Acorox 880. A synthetic grease produced from synthetic alkyloaromatic oil with aluminium-based thickener was also used.

3 RESULTS AND DISCUSSION

All results are presented in a graphical form as plots of modulus of elasticity, G, or damping coefficient, Q^{-1} as a function of temperature, T (see Figs 1–10). Figure 1 shows the results for the ceramic sample covered by the lithium-based grease containing 3% graphite additive. For com-

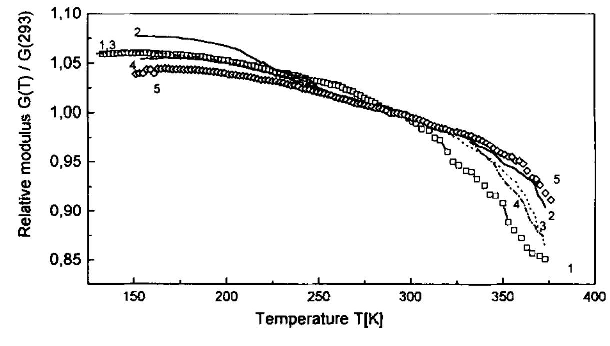

Fig. 2. Relative modulus as a function of temperature. (1) Ceramic without film of lubricant; (2) ceramic with deposited film of lithium-based grease containing 3% of graphite additive; (3) the same as (2) but after heating to 570 K; (4) the same as (2) but after heating to 620 K; (5) the same as (2) but after heating to 720 K.

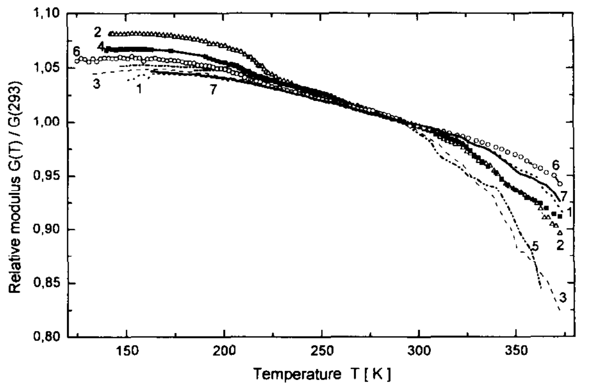

Fig. 3. Relative modulus as a function of temperature for ceramic. (1) (3) (5) (7) Without film of lubricant; (2) with deposited film of lithium-based grease plus 2% of extreme pressureadditive; (4) with deposited film of lithium-based grease; (6) with deposited film of lithium-based grease plus 3% molybdenum disulphide additive.

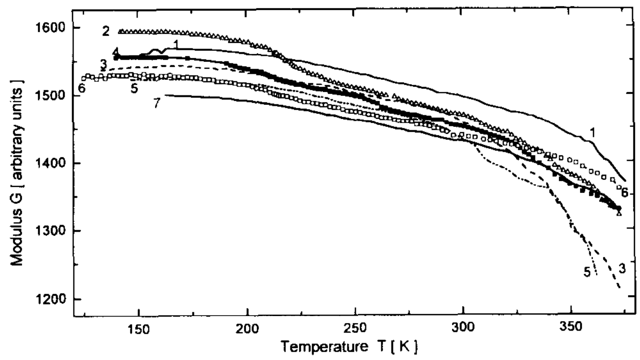

Fig 5. Elastic modulus G (in arbitrary units) as a function of temperature for ceramic. (1)(3)(5)(7) Without film of lubricant; (2) with film of lithium-based grease plus 2% of extreme pressure additive; (4) with film of lithium-based grease; (6) with film of lithium-based grease plus 3% of molybdenum disulphide additive.

parison, the changes in modulus of elasticity with temperature for the mild steel sample and the mild steel sample covered by the lithium-based grease loaded with 3% graphite are also shown. At low temperatures, the modulus of elasticity increases as a result of grease application for both the ceramic and steel. For both materials a clear change in elasticity modulus takes place in the range of temperatures 210–220 K which corresponds to the solidification temperature of base mineral oil. However, the increase in elasticity modulus for ceramic material is not as sharp as that for steel and amounts to 2–3% only. Similar changes in elasticity modulus are observed for ceramic material covered with synthetic grease, the only difference being that in this case the changes take place at a temperature several degrees lower. Again, this temperature corresponds well to the solidification temperature (182 K) of the synthetic grease. Most pronounced increase in G (~ 3%) occurs for extreme pressure and graphite additives.

At higher temperatures, where in the case of steel a second strong increase in elasticity modulus takes place, no increase in G takes place; on the contrary, a small decrease (~1%) is observed as compared to the ceramic sample without grease film. If the changes in G are considered together with the results of damping coefficient measurements (see Figs. 6–8) then the reason for such a small increase in G will be found. In the spectrum of Q^{-1} for ceramic material there is only one maximum and in the spectrum of the steel sample there is a second maximum present (see Figs 6 and 8). It is obvious that in ceramic material the mechanism responsible for damping in the steel at a temperature between 260 and 270 K is not operating and, therefore, there is no increase in G associated with this mechanism during cooling of the system.

Both the 2–3% increase in elasticity modulus, G, for ceramic material covered by grease film, as well as the occurrence of a maximum in the damping spectrum, systematically decay when the temperature during the heating-up cycle is sufficient to

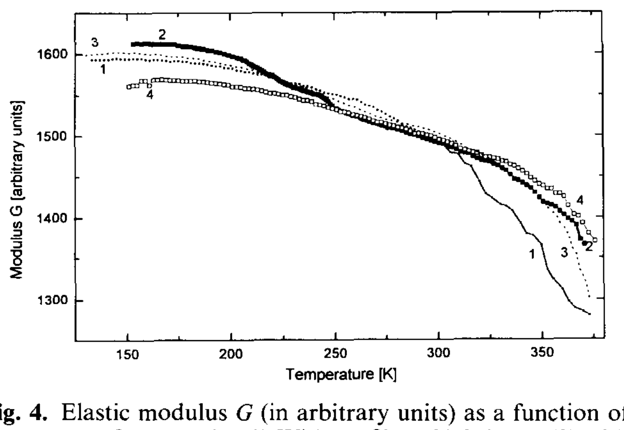

Fig. 4. Elastic modulus G (in arbitrary units) as a function of temperature for ceramic. (1) Without film of lubricant; (2) with film of lithium-based grease plus 3% graphite additive; (3) the same as (2) but after heating to 573 K; (4) without film of lubricant but after heating to 673 K.

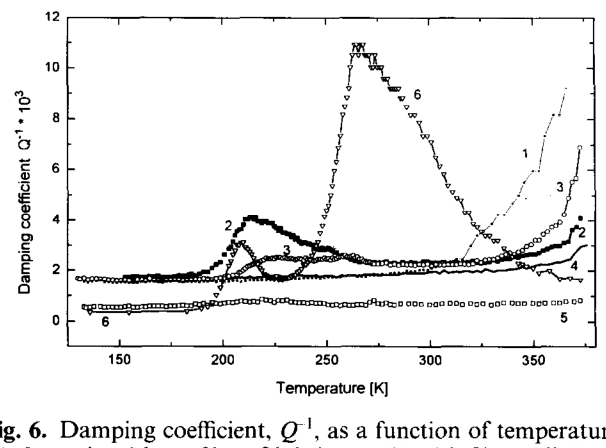

Fig. 6. Damping coefficient, Q^{-1}, as a function of temperature. (1) Ceramic without film of lubricant; (2) with film of lithium-based grease plus 3% graphite additive; (3) the same as (2) but after heating to 573 K; (4) the same as (2) but after heating to 473 K; (5) mild steel without film of lubricant; (6) mild steel with film of lithium-based grease plus 3% graphite additive.

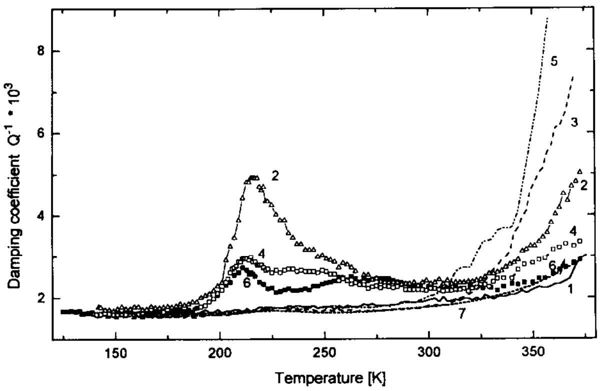

Fig. 7. Damping coefficient, Q^{-1}, as a function of temperature for ceramic. (1)(3)(5)(7) Without film of lubricant; (2) with film of lithium-based grease plus 2% of extreme pressure additive; (4) with film of lithium-based grease; (6) with film of lithium-based grease plus 3% of molybdenum disulphide additive.

cause degradation of the grease (see Figs 2, 4, 6, 8 and 9). However, after heating up the sample in vacuum of 10^{-3} torr to 673 K and maintaining it under such conditions for 30 min, the values of both elasticity modulus and damping coefficient return to initial magnitudes characteristic for samples without grease film. Besides, in the spectrum of $G(T)$, as well as $Q^{-1}(T)$, there are no sudden changes in the magnitude of those two parameters.

During multiple heating of the same ceramic sample covered by a thin film of lithium-based grease it was found that a systematic decrease in the elasticity modulus takes place. Figure 5 (curves 1 and 7) shows that the decrease is approximately 6%. This can be interpreted as a manifestation of continuous increase in the level of microcracks. Although studies using internal friction method are regarded as non-destructive, nevertheless, in the case of brittle materials, such as ceramics, they can lead to the propagation of existing surface microcracks which were produced either by processing[4] or during multiple heating-up cycles (thermal

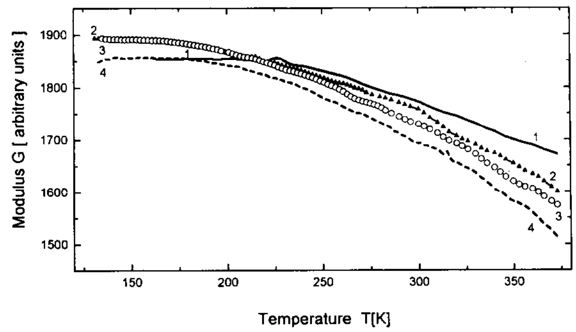

Fig. 9. Elastic modulus G (in arbitrary units) as a function of temperature for ceramic. (1) Without film of lubricant; (2) with film of synthetic lubricant; (3) the same as (2) but after heating to 573 K; (4) the same as (2) but after heating to 673 K.

stresses). Another reason for increased level of microcracks could be chemical adsorption.[5] This is particularly important in the case of the extreme pressure additive used during studies reported here. Close examination of Fig. 5 (curves 1, 2 and 3) reveals that this is indeed the case. The most pronounced decrease in G was observed for the ceramic sample covered by a film of grease containing EP additive. EP additive has a polar nature, while grains of Al_2O_3 are ion crystals, therefore, strong surface interactions, especially at the locations of existing structural imperfections, are quite possible. Interactions of this type usually result in breaking the surface bonds and, as a consequence of that, creation of permanent surface defects.[5]

All the results presented here point to the importance of the thin surface films for the elastic and damping properties of solid bodies. The exact mechanism responsible for the modification of elastic and damping properties of ceramic materials by surface films is not yet known, although, it is reasonable to assume that the source of observed phenomena is located at the interface between the film and surface of a solid body.

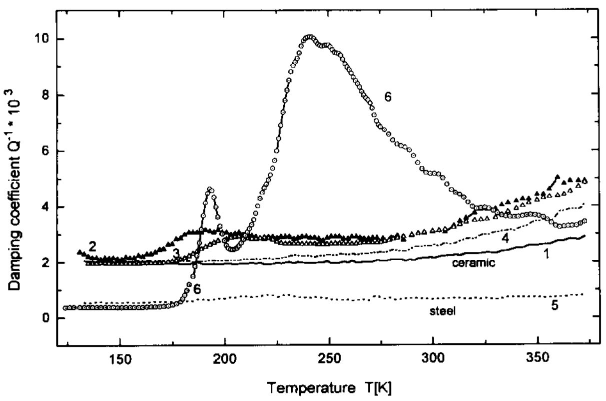

Fig. 8. Damping coefficient, Q^{-1}, as a function of temperature. (1) Ceramic without film of lubricant; (2) with film of synthetic lubricant; (3) the same as (2) but after heating to 573 K; (4) the same as (2) but after heating to 673 K; (5) mild steel without film of lubricant; (6) mild steel with film of synthetic lubricant.

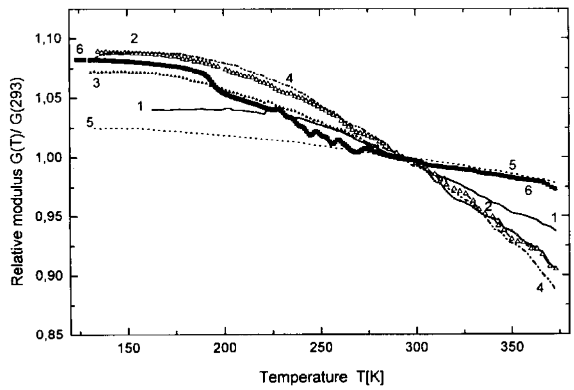

Fig. 10. Relative modulus as a function of temperature. (1) Ceramic without film of lubricant; (2) ceramic with film of synthetic lubricant; (3) the same as (2) but after heating to 573 K; (4) the same as (2) but after heating to 673 K; (5) mild steel without film of lubricant; (6) mild steel with film of synthetic lubricant.

4 CONCLUSIONS

The results of studies presented here could be used to support the following conclusions:

1. Elastic modulus and damping coefficient are affected by the film of lubricant deposited on the surface of ceramic material.
2. Extreme pressure additives proved to be especially effective in reducing modulus of elasticity of the material studied.

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