Multilayer Thermally Insulating Ceramic Contacts

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Stacks consisting of a number of thin layers of alumina (Al_2O_3) with thickness of either 1.0 or 0.65 mm were compressed in a direction normal to the individual layers under loadings of up to 1.2 MN/m^2. Three modes of elastic movements were encountered: a layer-flattening region at low loads ($P/M < 5 \times 10^{-5}$), followed by a surface waviness region under increasing pressure, and finally an asperity region ($P/M > 8 \times 10^{-5}$). Some hysteresis in the compression and the thermal resistance (the latter being measured in a vacuum of better than 1×10^{-4} Torr) characteristics were observed during the loading/unloading cycles, due to the poor recovery of the buckle in the layers. Thermal resistances per interface predicted by a mathematical model using parameters obtained from surface analyses showed good agreement with the experimentally obtained results. The ceramic surface was found to deform elastically, as predicted by plasticity index and surface analysis investigations.

Nomenclature

A_n = nominal area of contact between two surfaces, m^2
a = radius of a contact spot ($\bar{a}$ denotes the mean value), m
b = mean radius of the heat flow channels, m
E = Young's modulus of a contacting specimen, N/m^2
E' = effective elastic modulus, N/m^2
$g()$ = constriction alleviation factor
I = number of layer-to-layer interfaces in the considered stack of solid laminations
k = harmonic mean thermal conductivity of the bulk material, W/mK
l = thickness of each layer, m
M = microhardness of a surface, N/m^2
N = total number of contact planes in a stack, including those between the flux meters
n = total number of microcontacts in the nominal contact area
P = mechanical loading applied orthogonally to the contact interface, N/m^2
Q = rate of heat flux, W
R = thermal resistance of the pressed contact, K/W
R' = thermal resistance per contact of unit nominal area, m^{2}K/W
t = normalized separation between the mean planes of the contacting surfaces ($= u/\sigma$)
u = mean plane separation, m
X = number of ceramic layers
β^* = correlation distance, m
ΔT = temperature difference across the stack, K
λ = plasticity index of the microcontacts
ν = Poisson's ratio for the material of the considered contacting specimen
σ = root-mean-square (rms) surface roughness, m
$\phi(t)$ = normal probability function
$\psi(t)$ = normalized arithmetic-mean surface slope

Subscripts

B = bulk material
LL = layer-to-layer assembly
ML = flux meter-to-layer assembly
TOT,N = total for a stack of $(N - 1)$ layers

Introduction

TRADITIONAL superinsulations normally consist of an optimal number (per unit thickness in the direction of the expected heat flux) of thermally floating (i.e., not in pressed contact with one another), low-emissivity layers. Such low-effective thermal-conductivity structures have been widely used in cryogenic engineering, the individual layers being either metal foils interspersed with paper layers or a plastic sheet with ≈ 1-μ high-reflecting metal coating.[1] However, these structures are not designed to provide support, and their thermal resistance decreases catastrophically when subjected to mechanical load.[2] Thus, columns of metal disks have been exploited as mechanically strong thermal insulators.[3] The thermal insulation achieved is dependent upon the constrictional thermal resistances across each interface and on the number of interfaces formed.[4] Therefore, various metals, combinations of different metals, and/or metals coated with oxides have been employed in many trials.

A suitable alternative candidate material for the individual layers is a homogenous ceramic. The increasing interest in ceramics among design engineers stems largely from their ability to outperform many high-duty metal alloys and engineering plastics at very high temperatures.[5] Ceramics usually posses low thermal conductivities, relatively high hardnesses and stiffnesses, high melting points, and low densities.[6] Generally, ceramics are able to resist deformations at high temperatures, have high moduli of elasticity, and are more advantageous to use in compression, because their compressive strengths far exceed their tensile strengths.[7]

A knowledge of the thermal resistances of thin-layered stacks and their deformation behaviors under load, as mechanically strong thermal insulators, is vital for many traditional engineering applications (e.g., furnaces and refrigeration systems). However, the notable properties of ceramics lend themselves to high-cost, less traditional applications of these multilayer constructions. For instance, in screening and evaluating materials for the thermal protection system of the Space Shuttle, primary consideration was given to properties

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related to aerodynamic, structural, and thermal loading rather than to cost.[5] Moreover, the successes of spacecraft voyages are dependent upon the control of temperatures for the various components and surfaces.[8] Other areas of concern for thermal isolation include ablative heat shields, antenna struts, cryogenic storage compartments, and platform-mounted heat sources.[9] Thus, ceramic stacks could be utilized for the thermal isolation of critical, temperature-sensitive components. The aim of this experimental investigation was to study the behavior and the deformation of such stacks of ceramic thin layers under mechanical loads.

Thermal Resistance of Stacks of Thin Layers

Stacks of flat thin layers will provide effective thermal insulation, due to the high resistance to heat flow across each interface.[4,10] This high resistance is a result of the low ratio of real-to-nominal contact areas[1,3] caused by the microscopic irregularities and waviness holding the surfaces apart. However, the heat flow across an interface will encounter a resistance, due to the reduction in area through which solid conduction proceeds; thus, the lines of heat flux become constricted toward the asperity bridges. If the surfaces are buckled, then the ratio of real-to-nominal contact area is even less.[11]

The overall thermal insulation provided by a stack of layers depends on the resistance of each interface and the number of interfaces. To increase the thermal resistance per unit length of a stack, Thomas and Probert[4] recommended that 1) the thermal conductivity of the bulk material should be low; 2) each surface should possess a high surface hardness; 3) the material should have a large surface roughness; 4) each individual layer should be corrugated to give a smaller contact area and stiffness to inhibit its flattening underload; and 5) a large number of very thin layers should be employed. Inevitably, however, some of these conditions conflict with one another, for example, stiffness and the use of very thin layers. A further important property for a stack material is its yield strength to effective conductivity ratio, which should be high for mechanically strong (in compression) thermally insulating stacks.

Theoretical predictions concerning stacks of thin layers are far more complex compared with those for the single interface between semi-infinite bodies, where the deformations are significantly influenced by the substrates' mechanical properties. Stack deformation varies between primarily layer flattening at low loads to predominantly asperity deformation at high loads.[12] The magnitudes of such deformations depend on the number of layers, surface topographies, individual layer thicknesses, and the loading pressure. Hence, investigations regarding thin-layered stacks can only provide a qualitative indication of the way in which individual interfaces perform.[13]

Theoretical Analysis

Thermal Resistances of Pressed Contacts

The resistance that a heat flux encounters while passing across an interface occurs partly because it is constrained to pass through the microasperity contacts; these constrictions cause the heat to flow farther and laterally.[14,15] Where there are n such spots, each having a mean radius of $\bar{a}$, then the total resistance is given by

$$R = 1/2\bar{a}nk \tag{1}$$

where k is the harmonic mean of the thermal conductivities of the surfaces in contact, i.e.,

$$k = I/[\Sigma_1^I(1/k_I)] \quad \text{where} \quad I = 2 \tag{2}$$

To obtain a prediction for this resistance, an estimate of the overall number and mean size of the microcontact spots is required. By employing the theory of Tsukizoe and Hisakado,[16] the mean number of microcontacts can be found from

$$n = [\Psi^2 \pi t \phi(t) A_n]/8 \tag{3}$$

and the mean radius from

$$\bar{a} = 2/(\Psi \pi t) \tag{4}$$

However, in practice the contact resistance is less than that implied by Eq. (1), due to the interference between the heat flows passing through the neighboring asperity bridges. Hence, a constriction alleviation factor,[17] $g(a/b)$, is required; thus, the total contact thermal resistance for a single interface becomes

$$R = g(a/b)/(2\bar{a}nk) \tag{5}$$

where

$$g(a/b) = (2/\pi)\tan^{-1}[(b/a) - 1] \tag{6}$$

Surface Deformation

Onions and Archard[18] developed a plasticity index to describe the behavior of the contact between two surfaces of random roughness. This plasticity index λ is defined in terms of the rms value of the height distribution σ and the correlation distance β^*, which is associated with the major wavelength of the surface undulations. The plasticity index can be calculated from

$$\lambda = (E'/M)\cdot(\sigma/\beta^*) \tag{7}$$

where

$$E' = E/(1-\nu^2) \tag{8}$$

When the plasticity index is <0.7, then the probability for the occurrence of plastic deformations is very small.

Experimental Studies

Thermal Resistance Test Rig

A longitudinal one-dimensional heat flow rig was used for both mechanical and thermal tests. The rig consisted of a heater connected to a time-controlled power pack, two identical titanium flux meters, with the ceramic stack positioned between them, and a chiller. All of these components were

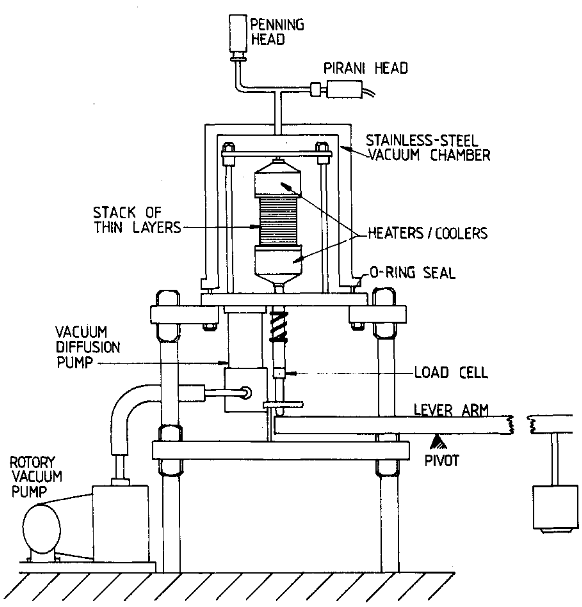

Fig. 1 Schematic diagram of the thermal contact resistance rig.

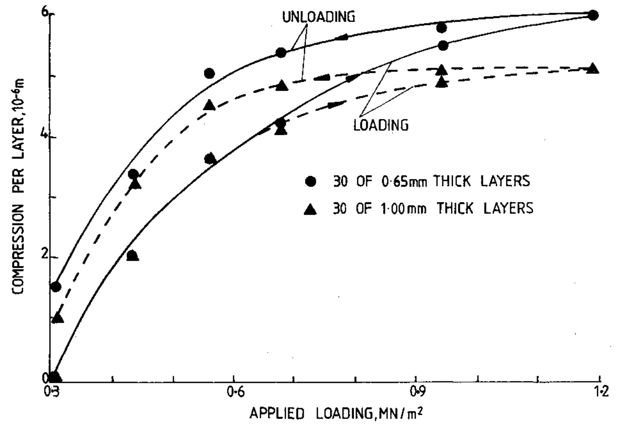

Fig. 2 Hysteresis effect of a stack of 30 disks; 0.65- and 1.00-mm-thick alumina layers.

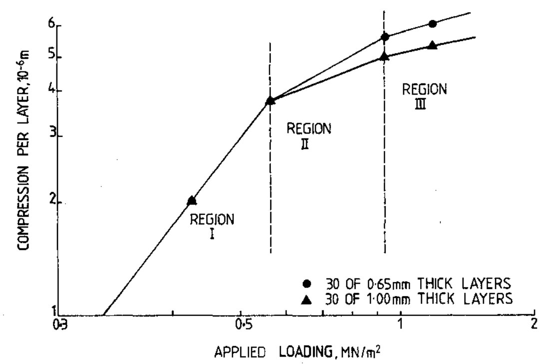

Fig. 3 Characteristic compression per layer with loading for a freshly assembled stack of 30 disks; 0.65- and 1.00-mm-thick alumina layers.

assembled in a vertical arrangement (see Fig. 1) and wrapped around with multilayer reflective superinsulation between the stack and the stainless steel vacuum chamber.

The total thermal resistance across the ceramic stack was determined by measuring the mean steady-state temperature drop between the opposite end faces of the flux meters as well as the heat flux crossing the interfaces, i.e.,

$$R = \Delta T/Q \tag{9}$$

The temperature difference ΔT was derived from thermocouple readings using a least-mean squares straight-line fit to the temperature/distance data for each flux meter. The heat flux Q was obtained from the axial temperature gradients in the flux meters and the temperature-dependent conductivity of the metal. A dial test indicator was used for the mechanical deformation measurements.

Specimens

Alumina (Al_2O_3) was chosen as the suitable ceramic material for the stack layers, the materials being coded ADS 96R (96% alumina), which is fine-grained, with a high alumina content. The thermal conductivity of the alumina is 21.75 W/mK, and its microhardness is 11.1×10^9 N/m². It also has a density of 3.98×10^3 kg/m³ and a specific heat of 778.22 J/kg K.

The 100-mm square alumina substrates were obtained from manufacturers in thicknesses of either 0.65 or 1.0 mm and used in an "as received" condition. However, these samples were cut into 25-mm-diam disks (i.e., same dimension as that of the flux meter) by using a diamond drill bit. During this process all surfaces were routinely cleaned, since ceramic particles could cause the specimens to shatter under pressure. Since the diamond drill bit could only produce a rough circular shape, the disks were given a more regular finish using a green grid grinder. The disks were subsequently thoroughly cleaned by being soaked in methanol, to remove any grease or contaminants that may subsequently affect the thermal and mechanical performance of the stack. Further cleaning was achieved by means of an ultrasonic bath containing distilled water, which removed any particles from the pores. This cleaning was imperative since a particle between the layers could have caused the material to shatter under compression. The specimen disks were allowed to dry for at least 48 h before they were tested. The interfaces of the titanium flux meters were also cleaned using methanol. Approximately 100 disks were prepared for each ceramic thickness, since the disks could not be reused after loading.

Surface Analysis Profilometer

Surface topographical measurements of the alumina layers were derived from traces obtained by employing a modified Talysurf 4 profilometer. This profile-tracing instrument incorporates an automatically controlled, three-dimensional relocating stage, as well as microcomputer-based data-handling and processing facilities.[19] The relocation stage consists of two moving tables: one that moves in a direction parallel to the locked stylus arm, and another, after completion of a trace,

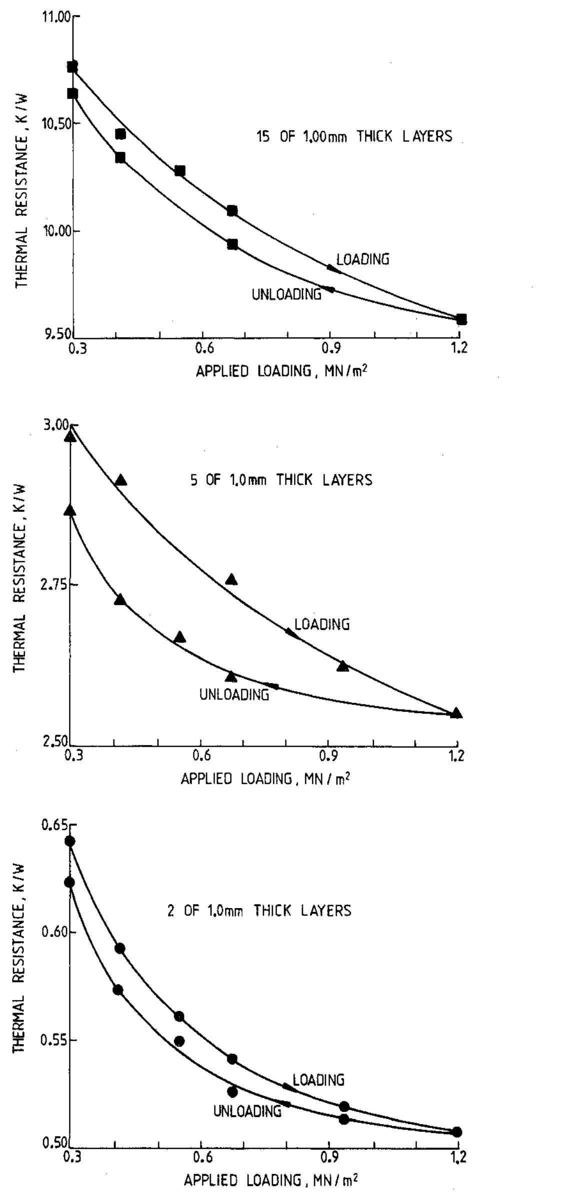

Fig. 4 Variation of the thermal resistance with the applied loading for a stack of 2, 5, and 15 alumina layers, each 1.0 mm thick.

that shifts a small, predetermined amount orthogonal to the arm.

Experimental Results

Typical examples of the compression characteristics resulting from the application of pressure to a stack of 30 disks, each having a thickness of either 0.65 or 1.0 mm, are shown in Figs. 2 and 3.

The total thermal resistances were obtained for stacks consisting of 2, 5, 15, and 30 layers of the two alumina specimens under a range of loading pressures in vacua (of better than 1×10^{-4} Torr). Figure 4 shows a typical example of these measurements. It was found that, during data reduction, compound errors arising from the experimental measurements result in uncertainty in the thermal contact resistance values of approximately $\pm 10\%$.

A stack consisting of $(N-1)$ layers will have a total of N contact planes, including the two extreme contacts with the flux meters and $I\ (=N-2)$ layer-to-layer interfaces. There will also be a resistance resulting from the bulk material R'_B, which is defined as l/k for each layer. The total resistance is given by

$$R'_{\mathrm{TOT},N} = 2R'_{ML} + (N-1)R'_B + (N-2)(R'_{LL})_{N-2} \tag{10}$$

Thus, for a single layer, $N=2$,

$$R'_{\mathrm{TOT},2} = 2R'_{ML} + R'_B \tag{11}$$

The resistance of an interface per unit area for a stack can therefore be deduced from the measured overall resistances and the bulk resistance of thin layers.[12]

Discussion of Results

Stacks of Thin Layers

During the mechanical investigations of alumina stacks, hysteresis in the behavior (a typical example is shown in Fig. 2) was detected for both layer thicknesses of the stacks with respect to the loading/unloading cycle. However, as expected, the stacks showed a large proportion of elastic recovery compared with stacks comprised of tin-coated steel.[13] This is due to the high-elastic moduli of the ceramic layers. Nevertheless, the stack composed of 0.65-mm disks showed larger compressions per layer than that of the 1.0-mm disks, because the thinner layers were less stiff and, hence, moved by larger amounts under similar loadings.

The relationship between the applied pressure and the compression per layer is shown in Fig. 3 for stacks of 30 layers of 0.65-mm alumina disks. The stack that was tested exhibited three characteristic regions of elastic movement: a rapid increase in the compression per layer with increasing pressure during low loads, followed by a transition at P/M of approximately 5×10^{-5}, at which there was a reduction in the increase in compression per layer for heavier loads, and, finally, a further reduction beyond a P/M value of approximately 8×10^{-5}. Similar observations were recorded for the stack of 1.0-mm-thick alumina disks. The initial linear characteristic (region I) was due primarily to the removal of the residual buckle from each layer, whereas in region II the effect of increasing the load was to enlarge the areas of the individual microcontacts and simultaneously to smooth out the waviness present in the surfaces. The final region (III) could be a result of either elastic or plastic deformation of the asperities, or a combination of the two. Probert and Jones[20] derived similar observations for metallic sheets, where they found that the main source of deformation was layer bending, whereas at high loads, asperity deformation predominated. However, since alumina has a high-elastic modulus, it is unlikely that there was any considerable plastic deformation of the asperities. Hence, the hysteresis observed for the stacks could be attributed to the absence of appreciable recovery in the buckle or possibly even due to the lack of recovery in the waviness. It was noted for all of the hysteresis plots that the accuracy of the experimental values for the compression per layer was within $\pm 10\%$.

Hysteresis was also observed in the thermal contact resistance with respect to loading/unloading cycles for the stacks (see Fig. 4). Each stack configuration showed a high proportion of elastic recovery during unloading, which supported the findings from the compression investigations.

As the load increased, the surfaces were brought into closer contact; hence, the number of microcontracts increased. Therefore, this reduced the thermal resistance in a direction normal to the stack, due to the increase in the thermal contact conductance across the interfaces. The steep decrease of thermal resistance may be attributed to the redistribution of the heat flow lines. However, for idealized solid-to-solid contact these lines converge toward the actual contact spots, thereby creating a constrictional thermal resistance. This phenomenon becomes more pronounced for stacks of thin layers because the heat fluxes are more narrowly constrained by the thinness of the individual layers. As the applied mechanical loading increases, the buckle flattens and then the surfaces conform according to their macroscopic topographies.

The thermal contact resistance per interface under increasing applied pressure was almost independent of the number of layers in the stack under similar loading cycles (see Fig. 5). The apparent variations in the resistance per interface for a particular number of layers are due to differences in the initial buckle of the individual layers.

Also exhibited in Fig. 5 is the relationship between the experimental thermal resistances per interface and the theoretical predictions derived by the theoretical model as summarized by O'Callaghan and Probert.[17] The latter showed reasonable agreement with the experimentally derived values. The predicted values were lower than those obtained from each stack, which could have been due to the occurrence of buckle and waviness of the surfaces, hence introducing an additional macroscopic resistance (i.e., the heat must flow through the

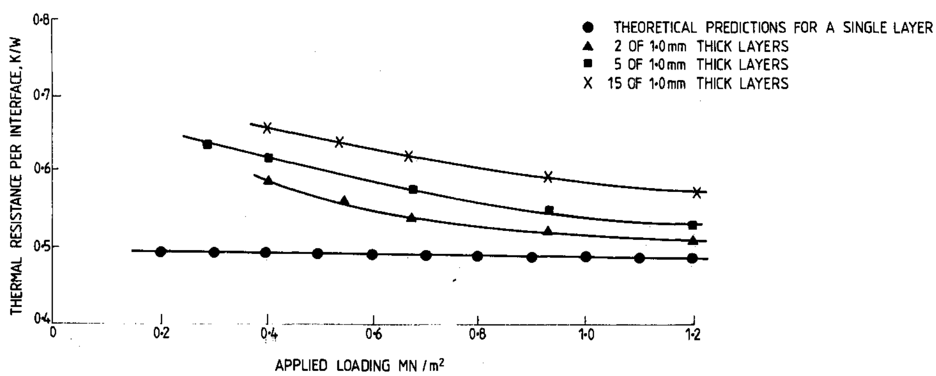

Fig. 5 Variation of the thermal resistance per interface with the applied loading for various stacks of thin layers.

buckle) and therefore providing a high thermal contact resistance. The data obtained could be represented by the following correlation per interface:

$$R = 0.6\ \exp(0.11X - 170P) \qquad (12)$$

This observation is reinforced by the more rapidly decreasing experimental resistances compared with the theoretical predictions, as the buckle and waviness were pressed out. Moreover, results from previous investigations of Probert and Jones[20] and Mikesell and Scott[3] indicate that the thermal resistance should be inversely proportional to the compression. However, Al-Astrabadi et al.[13] showed that such a conclusion only applied once the buckle was removed. Nevertheless, for the ceramic stack, Fig. 6 shows that after the buckle has been removed (region I), there are two regions of elastic movements (II and III), each of which could comply with such a relationship. These characteristics agree with the data of Fig. 3.

Surface Analysis

The investigations showed that the nominally smooth alumina surfaces exhibited a high density of asperities of low peak-to-valley heights. A rms roughness of 0.543 μ and a mean absolute surface slope of 0.0454 rad (2.6 deg), obtained from profile tracing, were used to predict the theoretical thermal resistance for a single interface.

A surface trace produced on a specimen, previously subjected to the loading, showed no visible deformation from the trace of a fresh specimen. Therefore, this suggests that the elastic movements experienced by the stack are more likely due to the absence of recovery of the surface buckle, rather than due to permanent deformation of the waviness and asperities. This is also supported by the consistency of the values of the surface parameters.

The value of β^* was derived to be 90.7 μ, and the plasticity index of the alumina was calculated to be 0.17. Thus, this plasticity criterion suggests that the alumina behaves in an elastic fashion. This prediction supports previous discussions regarding the behavior of the stacks of alumina layers and observations from the surface traces.

Further representations of the surface topography of the alumina appear in isometric plots. Figure 7 clearly exhibits the waviness of the alumina surface, which is also highlighted by contour plots of the surface.

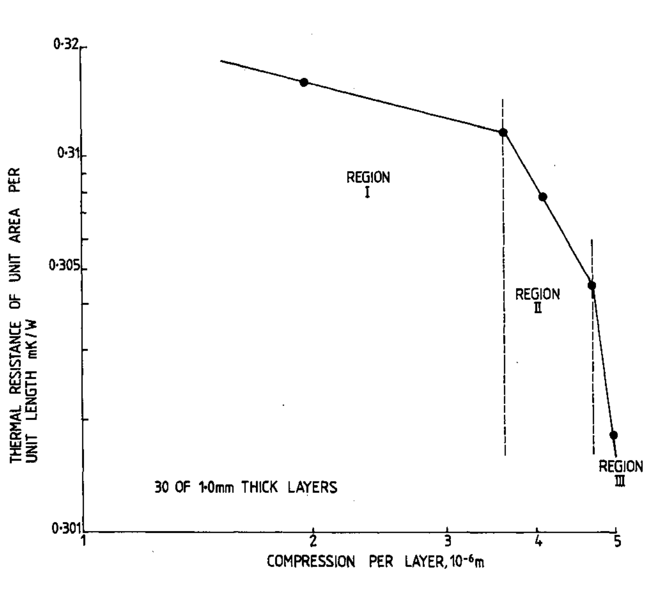

Fig. 6 Characteristic thermal resistance per unit area per unit length with the compression per layer for a stack of 30 disks; 1.0-mm-thick alumina layers.

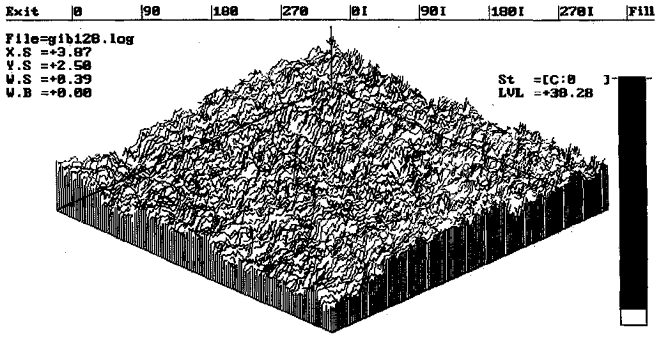

Fig. 7 Isometric plot of a typical surface of the alumina considered; the sampling interval is 3.865 μ.

Conclusions

The overall thermal insulation provided by a stack composed of alumina layers (Al_2O_3) is proportional to the number of interfaces. The mean thermal resistance per interface was 0.55 K/W at an applied pressure of 1 MN/m^2. This high resistance was due to the low ratio of real-to-nominal contact area ($\sim 1 \times 10^{-4}$ at an applied pressure of 1.1 MN/m^2) between adjacent alumina layers, the high degree of hardness of the ceramic surfaces, and the low-bulk thermal conductivity of alumina.

Previous studies concerned with metal layered stacks[21] showed that the bulk thermal resistances of the stacks were negligible compared with the contact resistances. However, this was not so with the alumina layers, due to the low thermal conductivity of the ceramic material. For example, the thermal resistance of the five-layered stack of 1.0-mm thickness was six times greater than a solid specimen of the parent material with similar overall dimensions.

Within the loading range $2.8 \times 10^{-5} < P/M < 1.07 \times 10^{-4}$, three distinct elastic-movement regions occurred. Initially, the load pressed out the buckle in the individual layers, followed by a region of an increasing number of microcontacts due to the smoothing out of the waviness present, and, finally, a transition to a region ensued where the effect of increasing the load was to enlarge the areas of individual microcontacts.

The alumina stacks showed some hysteresis with respect to the applied loading, due to the limited recovery of buckle encountered by the layers. However, the ceramic surface was found to move elastically in accordance with the indications resulting from the application of the plasticity index of Onions and Archard.[18]

The alumina surface possessed a large degree of waviness, which occurred in defined strips along the surface. This restricted the number of microcontacts and hence increased the thermal resistance.

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