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The mechanical behaviour of thin polyimide films on a silicon substrate under point loading

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A technique has been developed to measure submicrometre displacements of a stylus into a thin polymeric film bonded to a rigid structure under point loading. The technique was utilized to study the point-loading behaviour of a thin polyimide film (20 μm thick) spun cast on to a silicon wafer as a function of applied load. The displacement against time behaviour, along with the microscopic evidence, indicates that the deformation behaviour follows three regimes. The microscopic evidence suggests that below a contact stress of 0.1 GPa the material responds in a reversible manner. Progressive increase in the contact stress causes the material to deform in an elasto-plastic fashion. At high contact stresses material flow is exhibited along with crack formation at the boundary of the stylus. At contact stresses greater than 1.29 GPa, failure of the film is observed

1. Introduction

The increasing demands being placed on current interconnect technology in very large scale integrated circuit (VLSI) fabrication has spurred the development of interconnect systems that can support dimensions of less than a micrometre and multiple leads in a reliable fashion [1].

Since the first application of polyimide (PI) as an interlevel dielectric by Sato *et al.* [2], the use of thin-film PI in microelectronics has grown significantly [3–7]. Polyimides have many advantages over conventionally used materials with respect to planarization, thermal stability and processability.

During manufacturing and processing of VLSI, thin PI films experience a series of complex mechanical loadings, the most deleterious of which is compressive point loading. PI films are used as protective coatings at the I/O pins. During chip mounting the films are subject to point compressive loads. Also during the wire bonding procedure, point loads are applied over a short period of time. Therefore the behaviour of thin PI film on a rigid substrate, under compressive point loading, is a fundamental problem with significant technological implications in the field of microelectronic structures.

This paper will investigate the compressive point-loading behaviour of a thin PI film on a silicon substrate. Both time-dependent displacement behaviour and deformation mechanisms are examined over a range of applied loads.

2. Experimental procedure

2.1. Materials

A solution of polyamic acid in N-methylpyrrolidone was spun-cast onto 82 mm silicon wafers. The polyamic acid was derived from pyromellitic dianhydride (PMDA) and diamino-diphenyl ether (ODA). Each layer of polyamic acid was cured for 15 min at 85, 150 and 250° C in an N_2–H_2 atmosphere. Subsequent layers were spun on to these partially cured layers to achieve the desired thickness (each layer was approximately 5 μm thick). A final cure was performed for 30 min at 300° C and 30 min at 400° C. A final thickness of 20 μm was achieved.

2.2. Testing apparatus

An apparatus was designed and constructed to measure the load–displacement behaviour and simultaneously provide optical observation of the deformation mechanisms. A schematic diagram of the test apparatus is presented in Fig. 1. The apparatus consists of a tungsten carbide stylus (Semprex Corp., Campbell, California), machined to a given diameter and flatness, mounted in a cold-rolled steel platen. The platen was press-fitted on to a pair of linear bearings to facilitate smooth movement under an applied load. The loading of the stylus was achieved through a force applied to the platen. The platen was loaded by means of a lever arm (Fig. 1). A motor, set at a constant rate, controlled a retractable platform which supported the desired load. The force was transmitted through the lever arm to the platen. The entire apparatus rested on a stone base which itself was placed on isolation pads.

The silicon wafer was adhered to a test place using a thin coat of glycol terephthalate heated to 130° C. When slowly cooled to room temperature, the glycol terephthalate provided a rigid support for the silicon wafer during testing.

The displacement of the platen (and therefore the stylus) was monitored by a proximity gauge (Kaman Instrumentation Corp., Colorado Springs, Colorado). This non-contact measuring device allowed for

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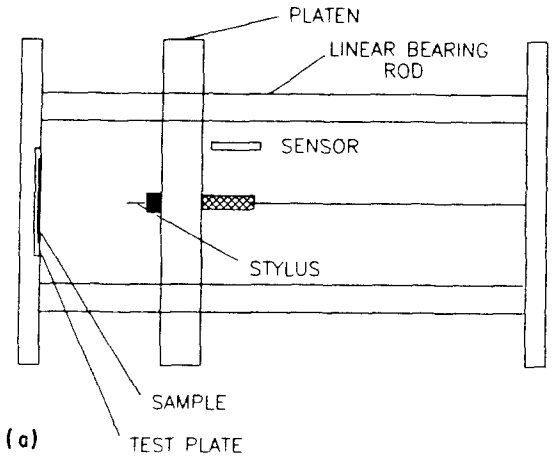


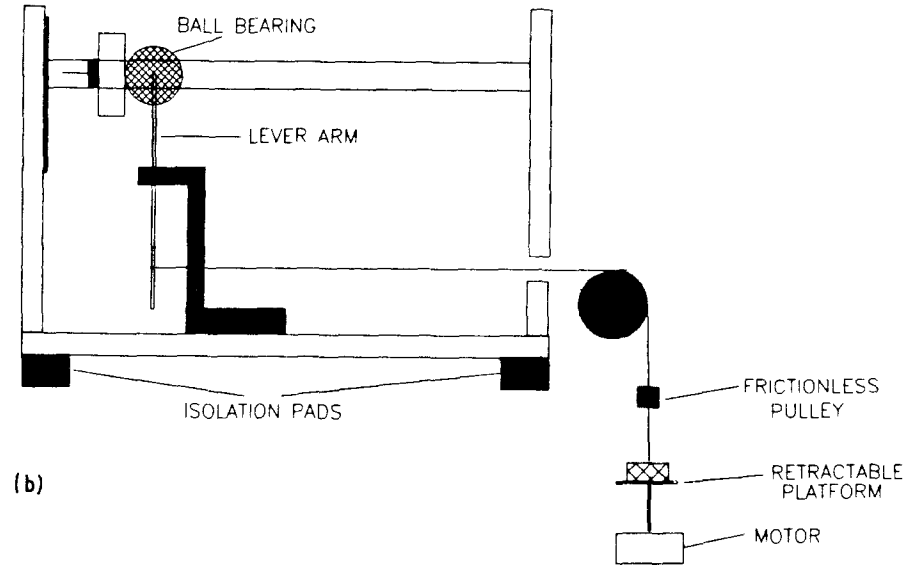


Figure 1 Schematic diagram of the testing apparatus designed to study the point-loading behaviour of a thin polymeric film on a rigid substrate. (a) Top view. The stylus, press-fitted into the platen, is used to load the film. (b) Side view. A lever arm and a pulley system is used to load the platen.

measurements of displacements to a fraction of a micrometre. For an input of 15 V, the proximity gauge was calibrated to output 3.9515 mV for every micrometre of displacement.

To estimate the level of friction in the linear bearings, a series of loads were applied until movement of the platen was obtained. A smooth movement of the platen was ensured at 20 g. The frictional force was therefore approximated as 20 g. To ensure proper contact during the initial loading, an optical microscope was mounted on the instrument. This feature allowed for real-time observation of the deformation process.

2.3. Testing procedure

A 100 μm stylus was used throughout this investigation. The films were tested at loads of 100, 500, 1000 and 2000 g at room temperature. A tare weight of 50 g was applied to ensure proper contact between the film and stylus. Displacement of the stylus was monitored as a function of time. The stylus displacement was corrected for displacements not associated with the film response. This correction was made for each loading condition by testing a portion of the wafer from which the film had been removed and subtracting this response from the data of the PI film. It should be noted that the tests done on the silicon wafer, after the PI film had been removed, exhibited only an instantaneous response with no time-dependent behaviour. The films were tested for one hour under a specified load, after which the stylus was unloaded. The net applied load was taken as the applied load plus tare load, minus the load associated with the friction in the bearings. All tests are reported in the form of a contact stress. The contact stress is defined as the net applied load divided by the cross-sectional area of the stylus.

2.4. Microscopic analysis

A Nikon SMZ-10 stereoscope was used to monitor and record the deformation processes during testing. An Aus Jena optical microscope and a Jeol 35CF scanning electron microscope were utilized to examine the film after unloading. The deformed zones within the thin films were examined under unpolarized and polarized light in reflection. Small sections of the wafer were cut to fit a two-inch (51 mm) SEM stage. A thin palladium film was evaporated on to the PI film before observation to enhance contrast in the SEM.

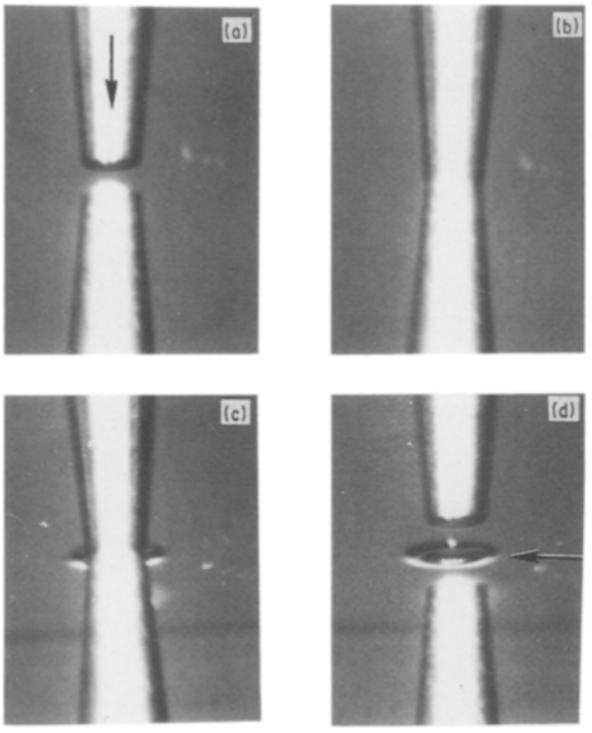


Figure 2 Series of optical micrographs detailing the point-loading phenomena for a 100 μm stylus at a contact stress of 0.66 GPa. (a) Stylus is positioned slightly above the film surface. Due to the polished nature of the substrate both a real and virtual image of the stylus are observed. The arrow in the micrograph indicates the direction of loading. (b) The stylus is loaded with a tare weight of 50 g and comes in contact with the film surface. (c) Taken approximately six seconds after loading. A ridge of material is seen around the probe. (d) Upon loading, the ridge is still present indicating that plastic deformation has taken place during loading.

3. Results

To elucidate the nature of the deformation processes, a series of photomicrographs were taken during the compressive point loading. In Fig. 2, optical micrographs of the loading sequence are presented for a film tested at a contact stress of 0.66 GPa. Fig. 2a shows the stylus positioned slightly above the film surface. The silicon wafer, being highly polished, acts as a mirror and produces the observed double image, one real and the other a virtual image of the stylus. Upon loading of the tare weight, the stylus is in contact with the polyimide surface (Fig. 2b). No deformation of the film is visualized at this magnification. The micrograph in Fig. 2c was taken at the onset of loading. A ridge was observed around the stylus indicating plastic flow of the film. After one hour the load was removed and the stylus withdrawn. Upon withdrawing the stylus the ridge observed during the loading was still present, indicating that irreversible deformation processes had taken place (Fig. 2d).

For a constant stress of 0.16 GPa there was an observed instantaneous displacement of 2.5 μm upon loading, followed by a gradual increase in the vertical displacement with time (Fig. 3). An optical micrograph of the film, taken after unloading, indicated that a clean punch-like deformation had taken place (inset to Fig. 3).

The vertical displacement against time curves for contact stresses of 0.66 and 1.29 GPa are presented in Fig. 4. Both exhibit behaviour similar to that observed for the film tested under a contact stress of 0.16 GPa. There was an instantaneous vertical displacement which increased sharply with the increase in load. It was also observed that as the load was increased there was a corresponding increase in the rate (slope) of the time-dependent displacement. The optical micrographs of the films, taken after unloading, show an increasing level of plastic deformation with increased load (inset to Fig. 4).

At a contact stress of 2.5 GPa, the displacement against time behaviour displays a large instantaneous displacement (approximately 80% of the initial thickness) (Fig. 5). Optical micrographs of the film sample indicate that the film has experienced extensive plastic deformation (inset to Fig. 5). Examination at higher magnification shows that the film directly under the stylus has been damaged. In Fig. 6 a close-up of the area directly under the stylus reveals that the integrity of the film has been destroyed.

Scanning electron micrographs confirm the

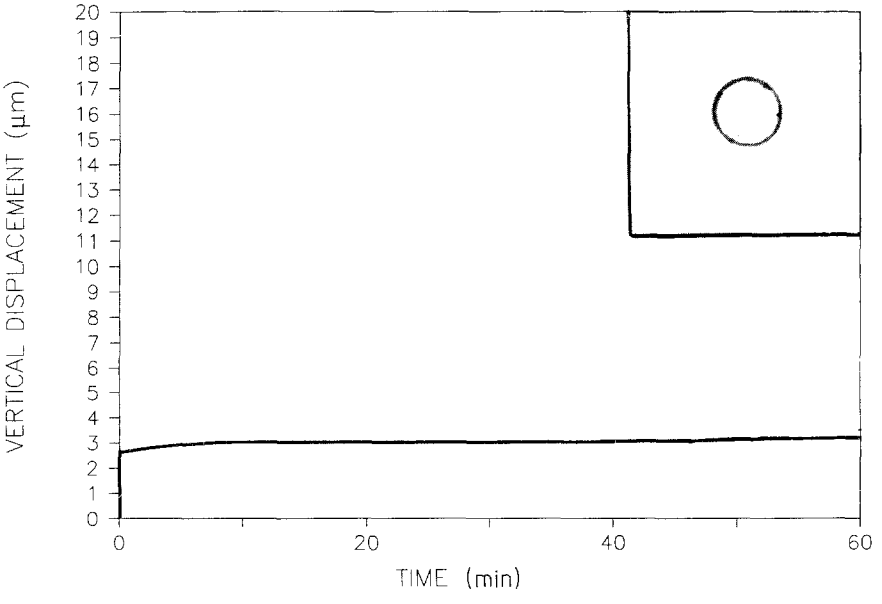


Figure 3 Displacement against time behaviour for polyimide (20 μm) film under a constant stress of 0.16 GPa (net applied load 130 g). Note that the curve consists of an instantaneous response followed by a time-dependent creep behaviour. Inset of graph is the optical micrograph of the film after unloading.

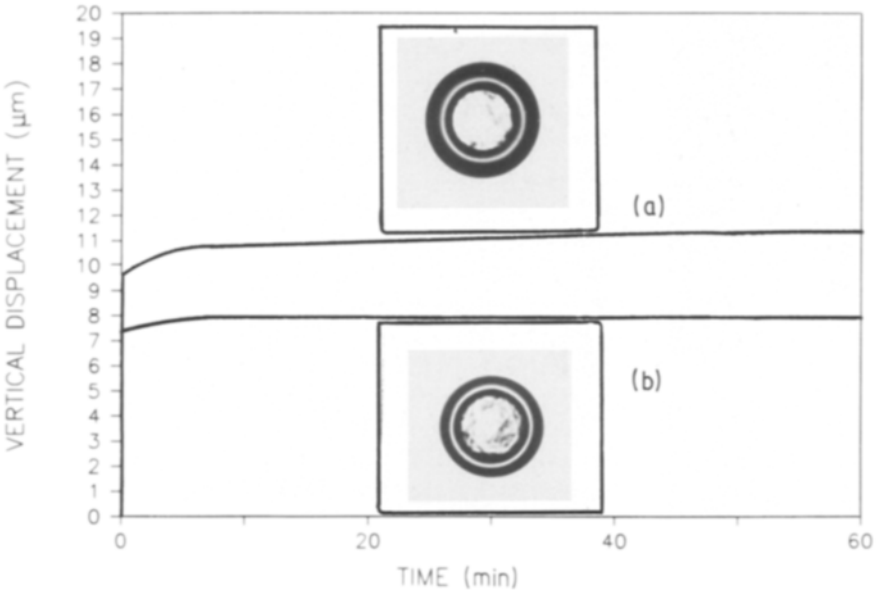


Figure 4 Displacement against time behaviour for polyimide (20 μm) film under constant stress of (a) 0.66 and (b) 1.29 GPa (net applied loads 530 and 1030 g, respectively). Note that the curves consist of an instantaneous response followed by a time-dependent creep behaviour. Insets of the graph are optical micrographs of the film after unloading.

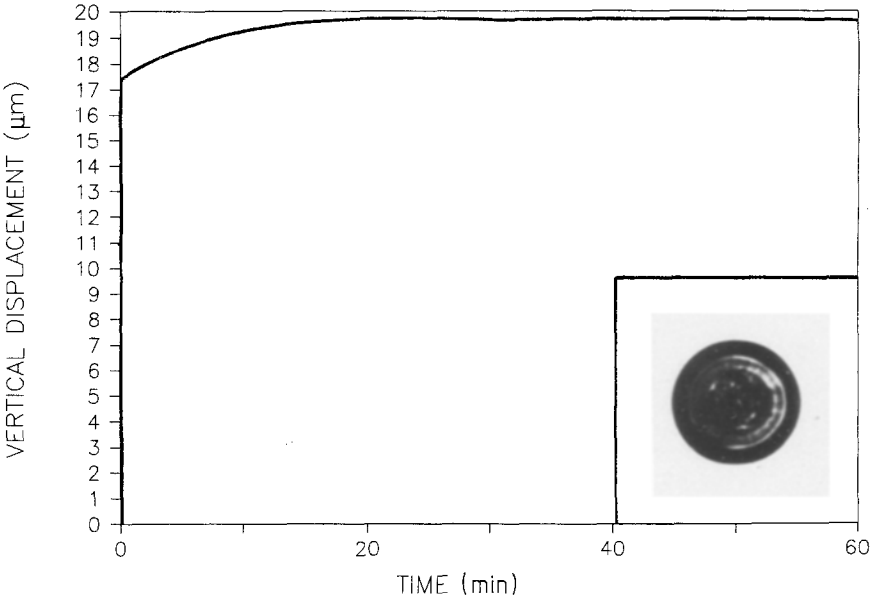


Figure 5 Displacement against time behaviour for polyimide film (20 μm) under a constant stress of 2.5 GPa (net applied load 2030 g). Note that the curve consists of an instantaneous response followed by a time-dependent creep behaviour. Inset of graph is the optical micrograph of the film after unloading.

observations made using the optical microscope. In Fig. 7 the SEM micrographs for films tested at contact stresses of 0.16, 0.66 and 1.29 GPa are presented. The micrographs indicate that at a stress of 0.16 GPa no plastic deformation has taken place. Only a clean punch-like deformation is observed. Below contact stresses of 0.1 GPa no irreversible deformation is detected in the SEM.

As the contact stress was increased two mechanisms of deformation were observed. First was the formation of cracks at the boundary of the stylus. Secondly, an increase in the plastic flow in the film was evident as the contact stress increased. At a contact stress of 2.5 GPa there was an observed change in the deformation behaviour (Fig. 8). The area under the stylus has been heavily damaged. The view given in Fig. 8 is slightly distorted due to the tilting of the sample in the SEM.

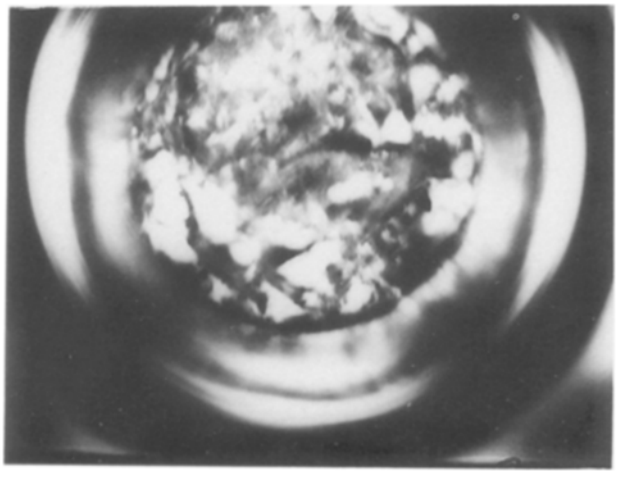
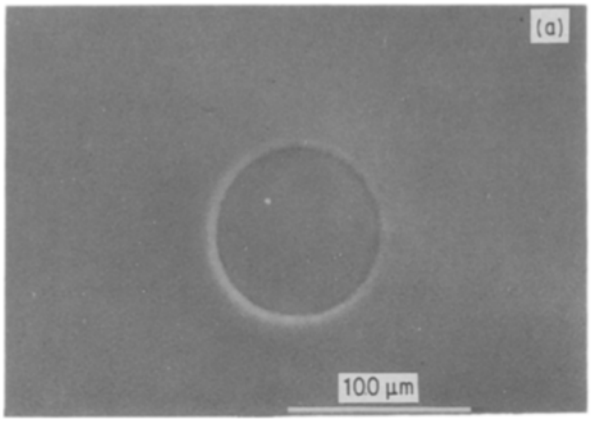
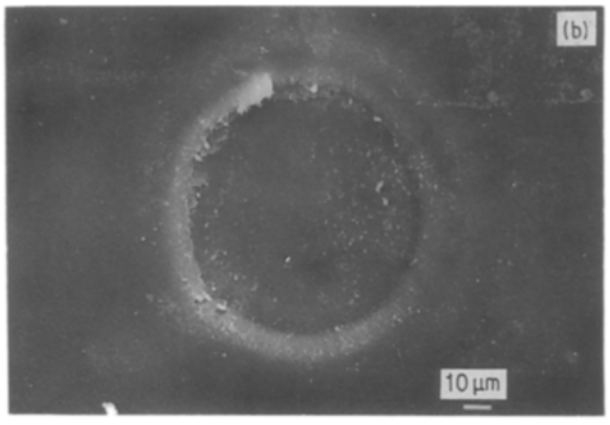


Figure 6 Optical micrograph of an area under the point of contact of polyimide film loaded at a contact stress of 2.5 GPa, showing that the integrity of the film has been destroyed.





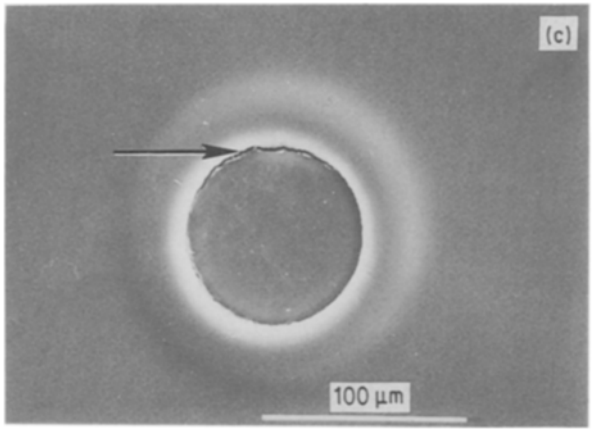


Figure 7 SEM micrograph of polyimide films deformed under compressive point loading at contact stresses of (a) 0.16, (b) 0.66 and (c) 1.29 GPa. Note increase in the plastically deformed material as indicated by the growing ridge around the contact area. At 1.29 GPa (c), a crack has formed at the base of the contact area (see arrow).

Surface traces of the 100 μm indentations were unsuccessful due to the lack of resolution available in placing the stylus on the film surface. However, surface traces could be obtained when a 500 μm stylus was used. In Fig. 9 the surface trace of a film tested at a contact stress of 0.15 GPa using the 500 μm stylus is presented. Due to difficulties in placing the stylus directly at the centre of the indentation, the trace was done on the upper half of the indentation (from X to X'). The trace clearly indicates that there is significant residual plastic deformation in the film.

4. Discussion

The deformation mechanism for thin PI film adhering to a rigid substrate appears to consist of three regimes. At low contact stresses (< 0.1 GPa), a reversible (elastic) response of the film predominates, with no detectable plastic deformation taking place. Above 0.1 GPa the film experiences increasing levels of plastic deformation. In Fig. 10 a normalized plot of the diameter of the plastic ridge, divided by the diameter of the stylus, is plotted as a function of the contact stress. The diameter of the plastic zone was measured using an optical microscope with the films under cross-polarized light. In Fig. 11 a representative optical micrograph of the films under crossed polarizers is presented. An asymptotic increase was observed, with a limiting value of approximately twice the diameter of the stylus. This plot of the extent of plastic deformation is a two-dimensional representation and does not take into account the material flow in the direction normal to the film surface. At higher contact stresses,

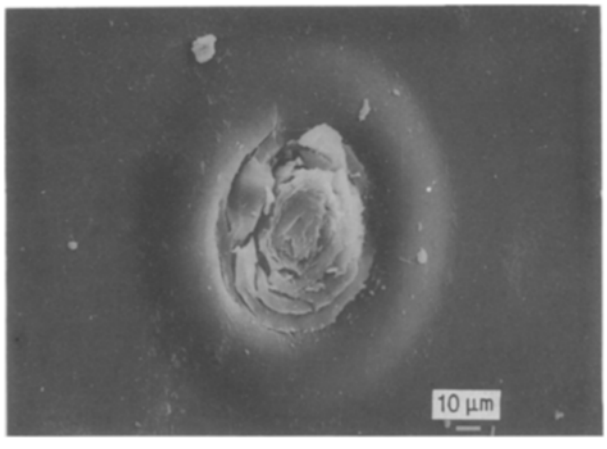


Figure 8 SEM micrograph of polyimide film deformed under compressive point loading at a constant stress of 2.5 GPa. The integrity of the film under the stylus contact area has been destroyed. Note that the film has been tilted in the SEM.

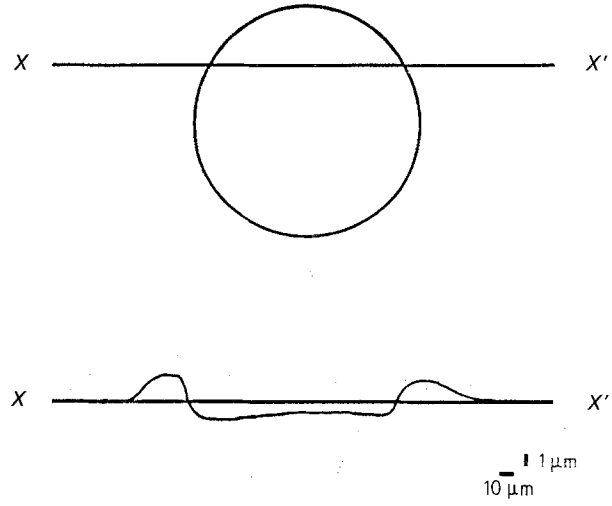


Figure 9 Surface trace of polyimide film tested at a load of 3000 g using a 500 μm stylus. The trace is from X to X' on the schematic diagram.

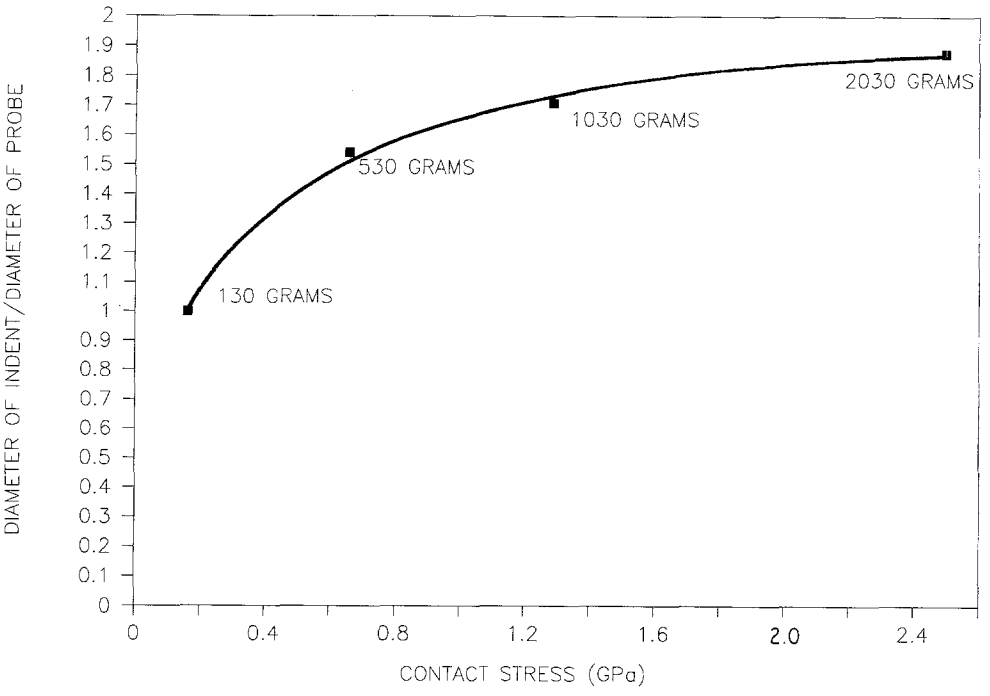


Figure 10 Normalized plastic zone plotted against the contact stress (also reported is the net applied load). The plastic zone approaches an asymptote at high contact stress/applied load.

crack formation was observed concurrent with the plastic flow.

Along with the changes observed in the deformation mechanism, the time-dependent response was found to be dependent on the contact stress. A creep component of the displacement behaviour can be determined by examining the difference between the instantaneous and final displacement after one hour (Fig. 12). By subtracting the instantaneous vertical displacement from the displacement–time behaviour the creep component of the displacement–time curves is elucidated (Fig. 13). At low contact stresses there is a small creep component, indicating that the majority of the observed deformation is reversible. As the contact stress increases the amount of creep detected also increases.

The SEM micrograph and surface trace show that there is considerable residual compressive strain in the film, as is suggested by the presence of ridges surrounding the point of contact. It has been observed that the ridges may be caused by debonding at the interface between the film and the substrate [8]. In our case there is no evidence from the optical micrographs to support this hypothesis (as is evident from the absence of interference rings under microscopic analysis in unpolarized light). The plastic flow observed about the stylus diameter is as predicted from the model of Yu *et al.* [9]. It should also be noted that the displacement of the film in the direction normal to the film surface at the stylus boundary is predicted in the asymptotic solution of the contact problem for a thin elastic layer [10].

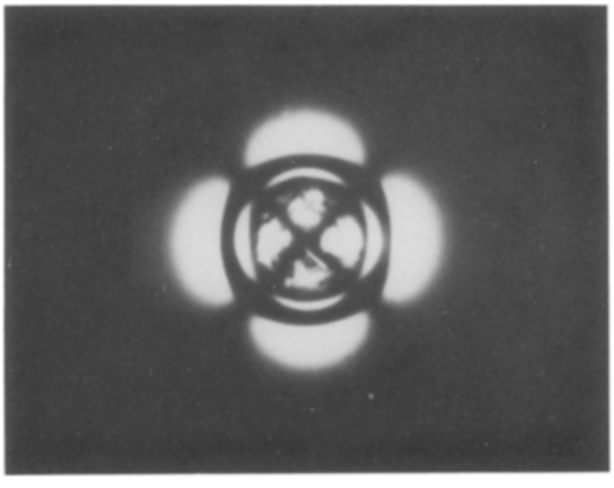


Figure 11 Representative optical micrograph taken under crossed polarizers of a polyimide film deformed under point loading at a constant stress of 0.66 GPa. Note the birefringent nature of the plastically deformed material around the point of contact.

5. Conclusion

An apparatus has been developed to measure submicrometre displacements of a thin polymeric film on a rigid substrate subject to point loading. The apparatus was utilized to study the point-loading behaviour of thin PI film on a silicon substrate. The displacement against time behaviour, along with the microscopic evidence, indicates that there are three deformation regimes present. The microscopic evidence suggest that below a contact stress of 0.1 GPa, the material responds in a reversible manner. Progressive increase in the contact stress causes the material to deform in elastic–plastic fashion. At high contact stresses material flow is exhibited along with crack formation at the boundary of the stylus. Finally, at contact stresses greater than 1.29 GPa failure of the film is observed.

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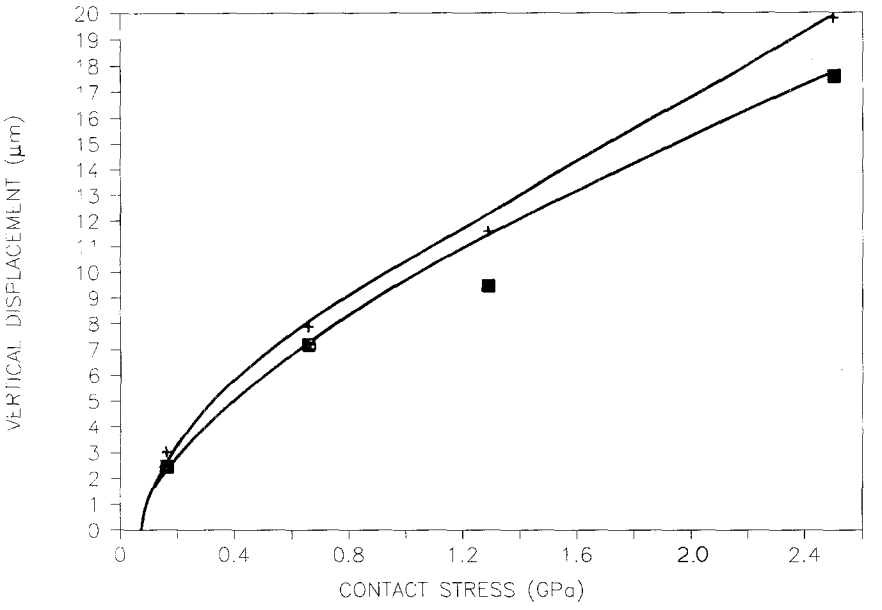

Figure 12 (■) Instantaneous and (+) final displacements as a function of the contact stress.

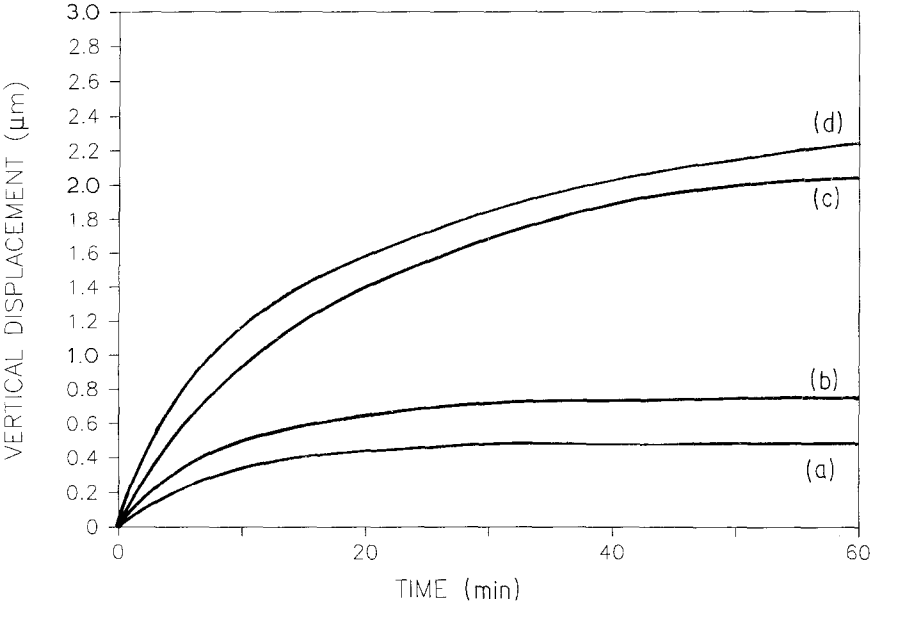

Figure 13 Time-dependent response of the polyimide films tested at constant contact stresses of (a) 0.16 GPa (130 g net load), (b) 0.66 GPa (530 g), (c) 1.29 GPa (1030 g) and (d) 2.5 GPa (2030 g).

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References

1. D. L. CROSTHWAIT, in Proceedings of University/Industry/Government Microelectronics Symposium, International Society for Hybrid Microelectronics, Starksville, Miss., May 26–28, 1981, x-1. IEEE, Piscataway, N.J. (1981).
2. K. SATO, S. HARADA, A. SAIKI, T. KIMURA, T. OKUBO and K. MUKAI, *IEEE Parts, Hybrid Packag.* **PHP-9** (1973) 176.
3. K. MUKAI, A. SAIKI, K. YAMANAKA, S. HARADA and S. SHOJI, *IEEE J. Solid State Circuits* **SC-13**(4) (1974) 462.
4. R. LARSEN, *IBM J. Res. Dev.* **24**(3) (1980) 268.
5. L. B. ROTHMAN, *J. Electrochem. Soc.* **127** (1980) 2216.
6. D. MAKINO, H. SUZUKI and N. KINJO, in Proceedings of University/Industry/Government Microelectronics Symposium, International Society for Hybrid Microelectronics, Starksville, Miss., May 26–28, 1981, x-48. IEEE, Piscataway, N.J. (1981).
7. A. M. WILSON, *Thin Solid Films* **83** (1981) 145.
8. P. A. ENGEL and D. D. ROSHON, *J. Adhesion* **10** (1979) 237.
9. H. Y. YU, M. A. IMAN and B. B. RATH, *J. Mater. Sci.* **20** (1985) 636.
10. V. M. ALEKSANDROV, *Appl. Math. and Mech.* **33** (1969) 49.

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