EVALUATION OF THE ELASTIC AND PLASTIC PROPERTIES OF Si_3N_4 BY DEPTH SENSING INDENTATION

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A high resolution instrument, called the nanomechanical probe, which permits continuous monitoring of load and depth of penetration during indentation and scratch tests has been developed at IAR/NRC. Its principle of operation, specifications and capabilities are discussed. Indentation tests on HIPed silicon nitride samples with densities ranging from about 82 to 99.7 % theoretical density have been performed. Hardness values for each sample have been calculated from load vs. depth plots using a new method involving the plastic work dissipated during the indentation process. The elastic properties of the samples have been determined from the slope of the unloading curve using the elastic punch model. The results are compared to those obtained by conventional methods.

INTRODUCTION:

Depth sensing indentation (DSI) tests have become very popular over the last few years because unlike conventional microindentation techniques, the load and depth of penetration or displacement are monitored during each test. This permits the calculation of hardness without the need to measure the diagonal of the indentation by optical or electron microscopy. Some of the many advantages to the DSI technique are: 1) better control of testing parameters, 2) faster data analysis, 3) smaller errors, 4) more data per test (a complete hardness vs. load plot can be obtained for each test) and 5) higher load resolution which is especially important for the testing of thin films and coatings. DSI has been used to calculate other properties of materials in addition to their hardness values. For instance, Loubet *et al* [1] and Doerner and Nix[2] suggested using the linear portion of the unloading curve to determine the elastic modulus of the material indented. The analysis is based on the elastic punch theory which was derived by Sneddon[3].

The DSI technique has also been used successfully to study the elastic and plastic properties[2,4], creep[5] and fatigue[6] of thin films and to evaluate fiber-matrix interfacial strength in composites[7].

Although the DSI technique appears to be very powerful in the evaluation of the mechanical properties of materials for which conventional methods can not be used, methods to calculate the various properties have not yet been standardized. In this paper, DSI tests were conducted on four HIPed silicon nitride materials using an instrument which was built in our laboratory. Load-depth plots produced at different maximum loads were used to calculate the Vickers hardness of the material using methods suggested by Doerner and Nix[2] and by Berriche and Holt[4]. In each case the results are compared to values calculated by using the measured diagonal of the indents. The elastic moduli of all four samples were determined using the elastic punch model.

EXPERIMENTAL PROCEDURE:

A table top DSI instrument with a load resolution of 5 mN and a depth resolution of 5 nm, called the nanomechanical probe (NMP), was built in our laboratory[8]. It consists of a high resolution xyz stage, a low voltage piezoelectric transducer (PZT) system, an indenter tip attached to a load cell and an optical system with a closed circuit device (CCD) camera and monitor. During the test, the area to be indented is first focused at 900 X magnification (on the monitor), using the z control of the xyz stage. This precise area of the specimen is then positioned underneath the indenter tip using a ball slide. The indentation test is carried out by moving the sample into the indenter tip with the PZT, driven in a closed-loop manner by a function generator at a frequency of 0.01 Hz (displacement control mode, triangular wave). During the test, a data acquisition system monitors the displacement and the load as the indenter penetrates into the sample (loading segment) and as it leaves the sample (unloading segment). The depth of penetration is extracted from the displacement by correcting for the compliance of the machine, which is 0.55 μm/N.

In the present study, the NMP was used with a standard Vickers diamond indenter to carry out indentation tests on four HIPed silicon nitride materials. Three of these materials were produced in our laboratory and are identified as EB4, EB9 and EB14 with densities of 82, 84 and 99.5 % theoretical (TD), respectively. Information on processing of these three samples along with their conventional hardness values were given in a previous paper[9]. The fourth sample is a commercially available HIPed Si_3N_4 of density 99.7% TD (known as NT154) which was purchased from Norton/TRW in the form of standard bend test bars.

All four samples were mounted in bakelite and mechanically polished. At least ten tests were conducted per sample at five different displacement amplitudes with each test producing one load-depth plot and one indentation impression.

RESULTS AND DISCUSSION:

(i) SEM Observations of the Indentations:

Two of the indentations produced on samples EB4 and NT154 are shown in Figures 1a and 1b, respectively. SEM pictures were taken of all the indentations and were used to measure the diagonal for each load. As expected, samples EB4 and EB9 with low densities displayed significant porosity; the indentations in these samples were accommodated by the collapse of porosity and no cracks were observed (Figure 1a). For the high density materials EB14 and NT154, there was extensive microcracking within the indentation impressions at all loads. In addition, cracks propagated from the corners of the indentations at loads higher than 1 N, as shown in Figure 1b.

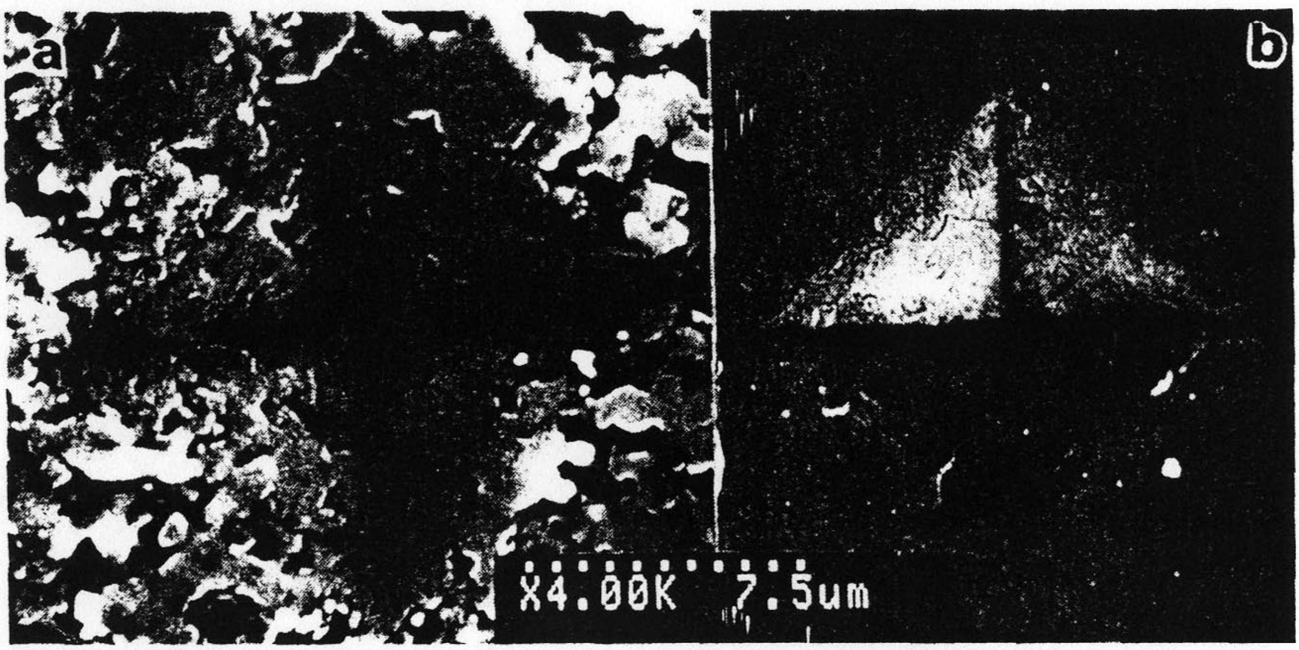

Figure 1: SEM micrographs of a) 1.19 N indentation in EB4 and b) a 1.73 N indentation in NT154.

(ii) Description of Load-Depth Plots:

A series of load-depth plots to different maximum loads are shown for sample EB4 in Figure 2. As can be seen, the load increases non-linearly with depth because of the increased contact area between the indenter and the material. The loading segments from different tests on the same sample all overlap indicating that the NMP has a good reproducibility and that the surface of the sample is homogeneous and smooth with respect to the size of the indentation. The unloading segments display an initial linear region due to elastic recovery followed by a non-linear region as the indenter loses contact with the sample.

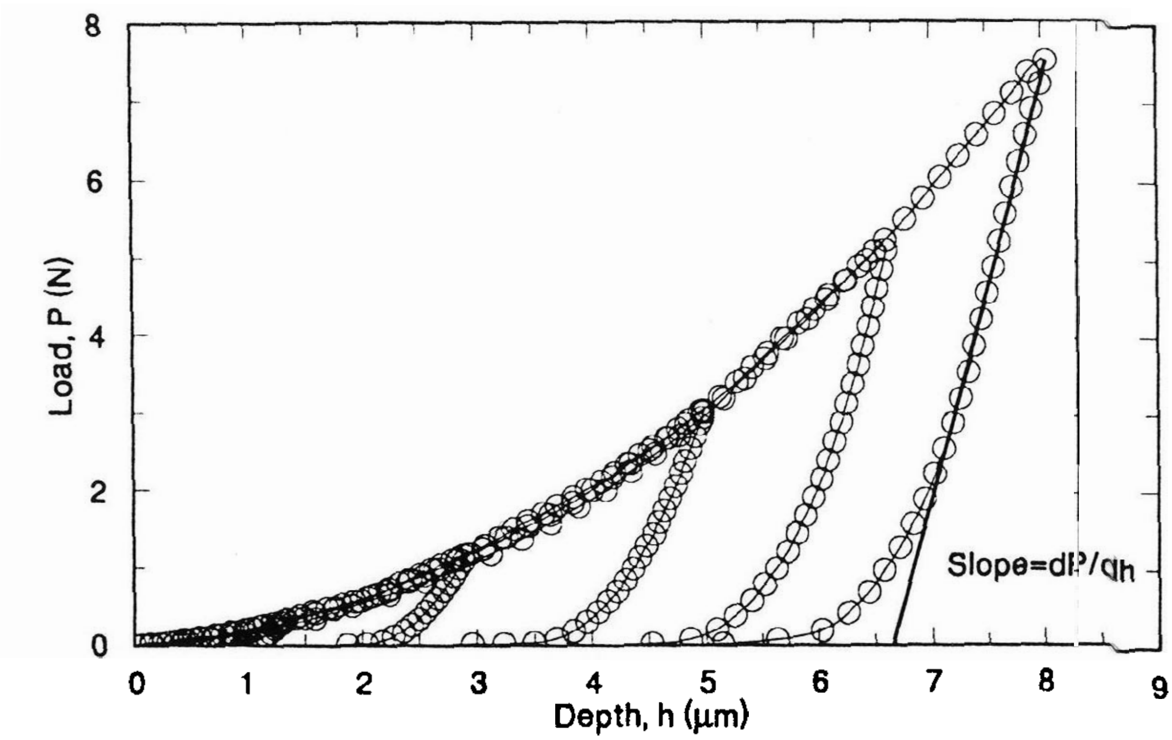

Figure 2: Load-depth plots at different maximum loads for sample EB4.

Figure 3 shows load-depth plots of two indentations made onto different regions of sample NT154 at the same small load of about 0.5 N. The impressions for these two tests were hard to find when this sample was examined with the SEM, and a magnification of 7,000 times was needed to determine the diagonals for conventional hardness calculations. Except for a small initial transient, the two load-depth plots overlap, indicating good reproducibility of the data even at very small loads. The small difference between the two loading segments at low loads was not generally observed for the other three samples. It may be due to the indenter coming into contact with porosity or a scratch.

For an overall comparison of the four materials studied, representative load-depth plots from tests carried out at the same displacement amplitude are included in Figure 4. Since the tests are displacement controlled, a higher load and a smaller depth were obtained for the high density samples NT154 and EB14.

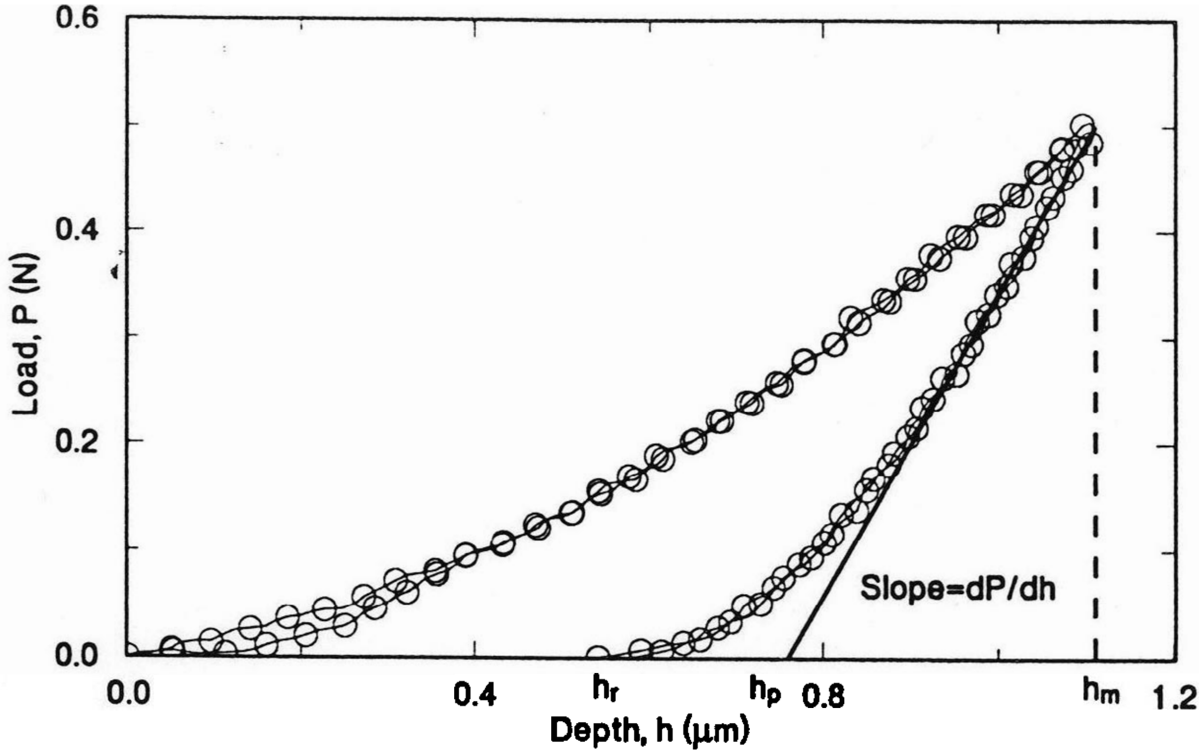

Figure 3: Two superimposed low load, load-depth plots for NT154.

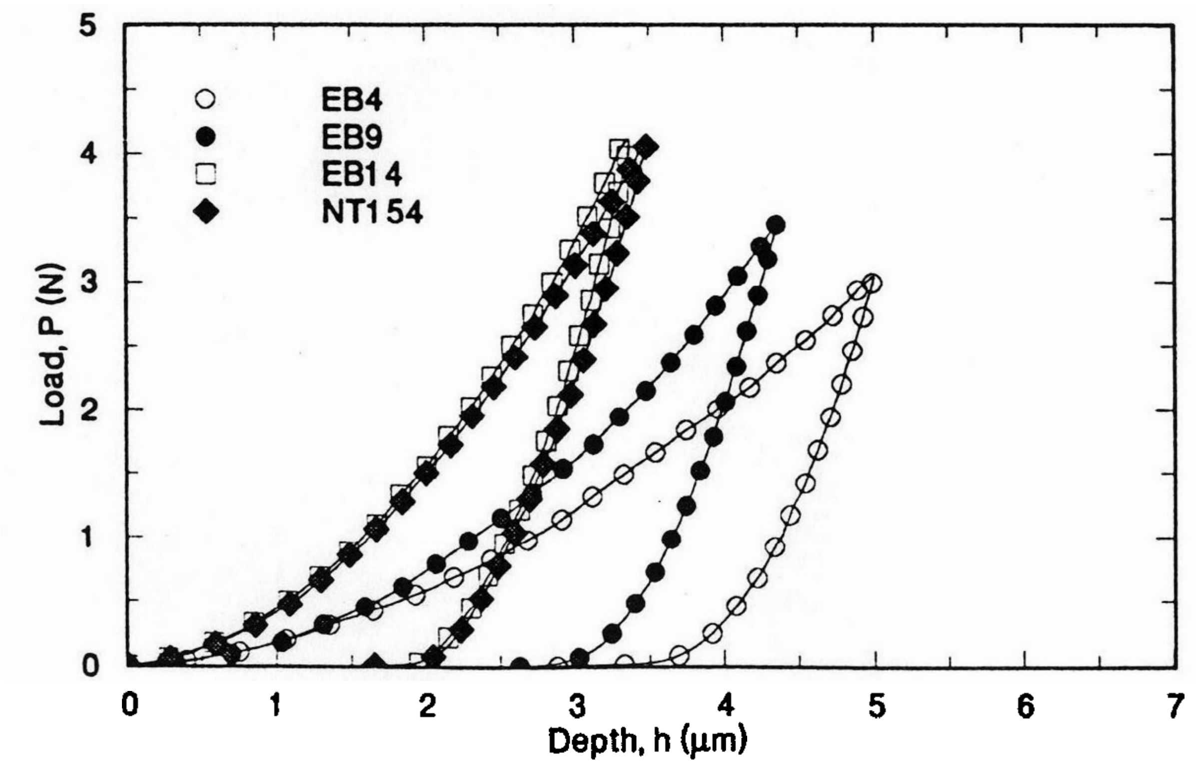

Figure 4: A comparison between load-depth plots obtained for samples EB4, EB9, EB14 and NT154 at the same total displacement.

(iii) Calculation of Hardness From Load-Depth Plots:

The theoretical relationship between the Vickers hardness HV in GPa, the load P in N and the diagonal d in μm is:

$$HV=1854\frac{P}{d^2}............(1)$$

Assuming ideal geometry, the ratio of diagonal to depth, h, is 7 for the Vickers indenter. So the hardness as a function of depth is given by:

$$HV=37.84\frac{P}{h^2}............(2)$$

Hence load-depth plots can be used to calculate the hardness of the material without the need to measure the diagonal of the indents.

Most of the previous reports on DSI testing have been carried out using Berkovitch indenters which are three sided pyramids with the same area to depth ratio as Vickers indenters. In these cases, the hardness has been defined as load over projected area, and is referred to as Meyer's hardness (HM). Referring to Figure 3, it can be seen that various depths can be defined, the maximum depth, h_m, the plastic depth, h_p, and the remnant depth, h_r. Doerner and Nix[2] found that using h_p gave the best agreement with conventional hardness values calculated using measured areas.

Another method to calculate the hardness from load-depth plots has been recently suggested by Berriche and Holt[4]. In this case, HM was defined as the plastic work, W_p, over the volume of the indentation produced, v. Since HM=HV/0.927, the following relationship can be derived for HV in GPa, W_p in μJ and h_p in μm:

$$HV=113.51\frac{W_p}{h_p^{\,3}}............(3)$$

The data acquisition and analysis programs for the NMP have been used to compute the hardness as a function of load based on equations (1), (2) and (3). Results for samples EB9 and NT154 are presented in Figure 5, and corresponding data were obtained for samples EB4 and EB14. Regardless of the equation used for calculating the hardness, Sample EB4 had the lowest hardness at all loads, followed by sample EB9. EB14, on the other hand, had hardness values which were comparable to those for the commercially HIPed Si_3N_4, sample NT154, at all loads. For instance, at a load of 4 N, the hardness calculated using equation (3) was 21.5 GPa for EB14 and 21.1 GPa for NT154.

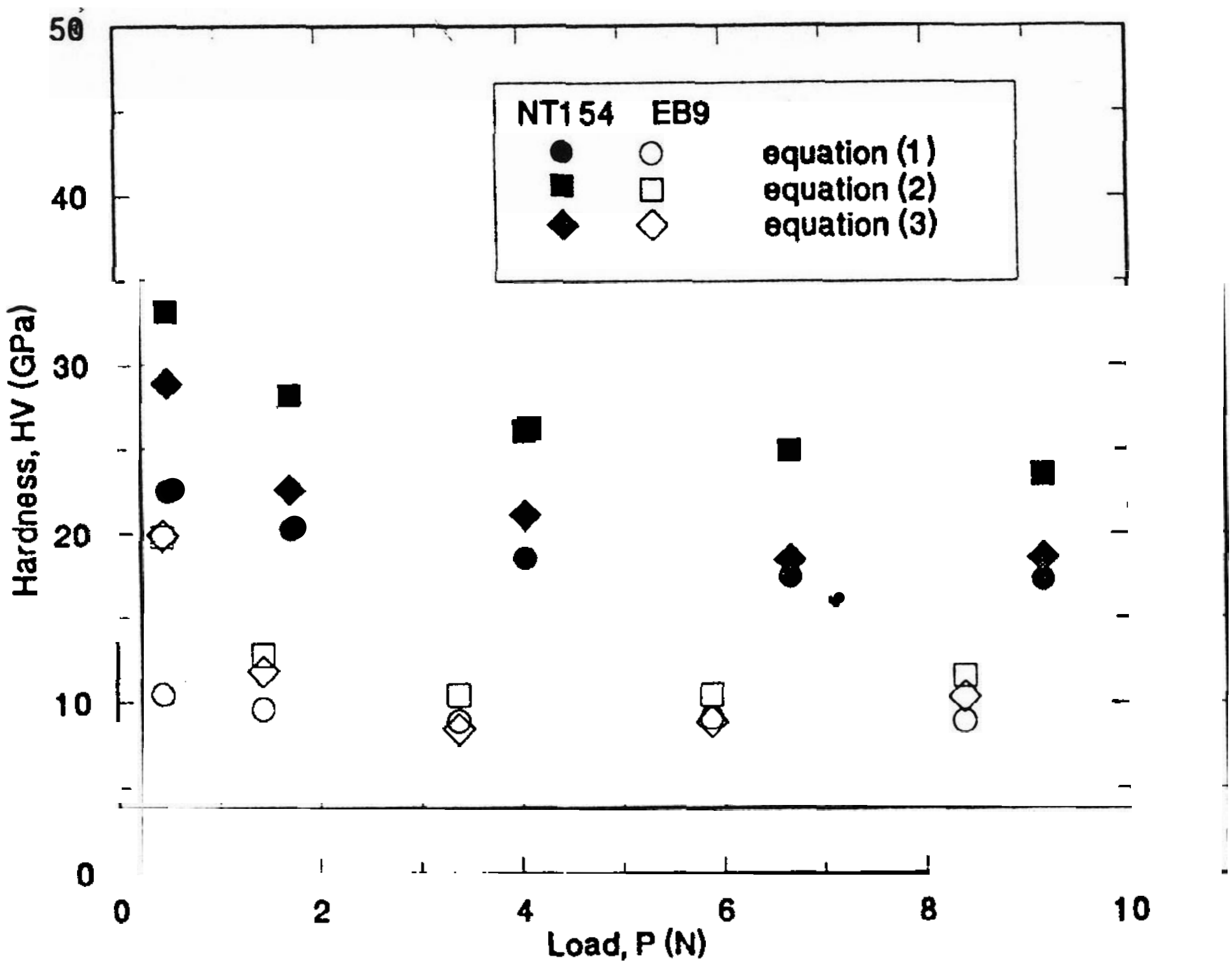

Figure 5: Hardness vs. load for specimens EB9 and NT154. Each point represents the average of two readings.

In general, hardness values obtained from equation (3) were very close to those obtained from equation (1). In all cases, better agreement is obtained between hardness values calculated using the diagonal and those calculated using the plastic depth when equation (3) was used instead of equation (2). The DSI technique to determine hardness values using the NMP is much quicker and easier than the conventional method which requires measuring the indentation size optically, or in the case of "nano-hardness" tests from SEM micrographs. The difference in hardness values as computed from the three methods is reduced at higher loads and is smaller for the low density samples. Finally, the hardness values obtained from tests using the nanomechanical probe are very close to values obtained for the same specimens using a Leitz microhardness tester[9].

(iv) Calculations of the Elastic Modulus:

As shown in Figures 2 and 3, the initial portion of the unloading curve is linear under all load ranges studied. The slope dP/dh of this region can be used to calculate the elastic modulus of the material indented[1,2,10]. The equation needed for the calculation has been derived from the analysis developed by Sneddon[3] for a flat cylindrical punch indenting an elastic half space (see ref. 10 for the derivation), and has the following form:

$$\left(\frac{1-\nu^2}{E}+\frac{1-\nu_0^2}{E_0}\right)\frac{dP}{dh}=2\sqrt{\frac{A_c}{\pi}} \quad \text{......(4)}$$

where, ν is Poisson's ratio of the sample being indented (for Si_3N_4 a value of 0.273 was provided by Norton/TRW Ceramics); E is the elastic modulus of the subject material; ν_0 is Poisson's ratio for the diamond indenter (0.27); E_0 is the elastic modulus of diamond (965 GPa) and A_c the contact area.

The calculated values of E obtained from equation (4) for samples EB4, EB9, EB14 and NT154 are presented in Figure 6 as a function of applied load. The values for NT154 are remarkably close to the value of 315 GPa provided by Norton/TRW Ceramics and this was further corroborated by tests performed on two NT154 bend test bars using the GrindoSonic resonant frequency method, which gave E values of 311 and 312 GPa. The results in Figure 6 show a decrease in the elastic modulus with a decrease in specimen density. Samples EB14 and NT154 with the highest density have the highest E values, followed by EB9 and then EB4. As expected, the calculated E values for any particular sample are virtually independent of the load.

These results confirm that the DSI technique can be used successfully to evaluate the elastic modulus of a small volume of material if Poisson's ratio is known. If the value for ν is not known, a value of 0.3 can be used in equation (4) without significant errors. This method has certain advantages over conventional methods to determine E (such as the resonant frequency method) in that the specimen geometry is not important. Furthermore, it is relatively easy, quick and inexpensive to perform.

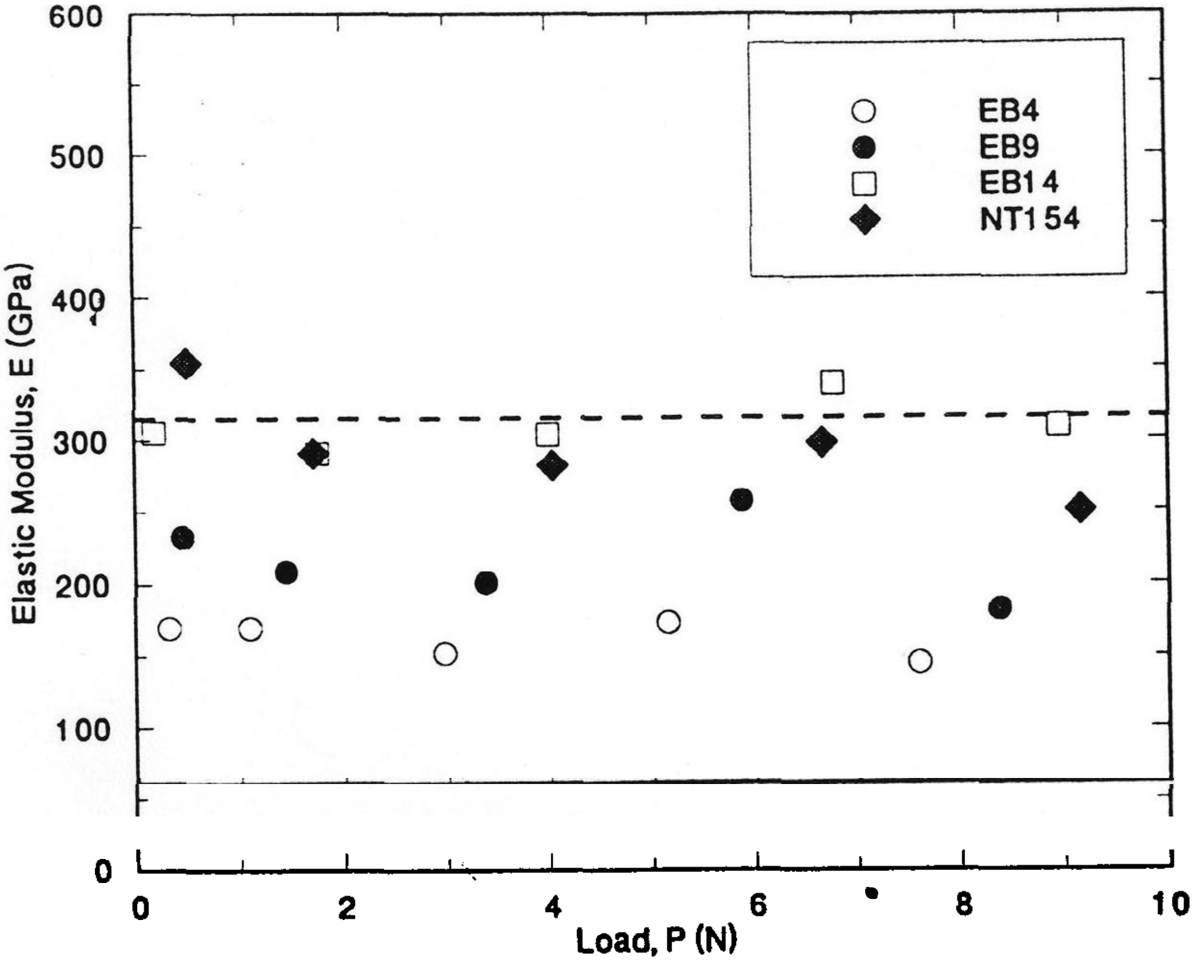

Figure 6: Elastic modulus vs. load calculated for four HIPed Si_3N_4 materials using equation (4). Each point is the average of two readings. The dashed line represents E=315 GPa as provided by Norton/TRW for NT154.

SUMMARY AND CONCLUSIONS:

1. A depth sensing indentation instrument built in our laboratory, called the nanomechanical probe, has been used successfully to evaluate the Vickers hardness and elastic properties of HIPed Si_3N_4 materials of various densities.
2. Two methods of calculating the hardness from depth of penetration were compared to the conventional method of measuring the diagonal size. The method which gave the closest agreement was that due to Berriche and Holt, which involves analysis of the plastic work determined from load-depth plots.
3. Hardness values obtained using the NMP are comparable to values previously obtained with a commercially available conventional hardness tester[9].
4. Load-depth plots can be used to determine the elastic modulus of small areas of materials or coatings. The calculated E values for NT154 were very close to values obtained by other methods.

5. Fully dense silicon nitride material produced in our laboratory had an E value slightly higher than that for NT154. E values were lower for the less dense samples. In all cases E was virtually independent of the indentation load.

ACKNOWLEDGEMENT:

This work was performed under IAR project JHQ00 in collaboration with Ceramics Kingston Céramiques Inc. with financial assistance from the Chief, Research and Development of the Department of National Defence, Ottawa, Canada (CRAD-DREP FE 144689 - NAE01). Mr. Andre Van Leuven of J. W. Lemmens Inc. kindly performed the GrindoSonic resonant frequency tests.

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