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MICROMECHANICAL CHARACTERIZATION OF NEAR-SURFACE LAYERS

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This paper reviews several techniques available to the experimenter to characterize the mechanical properties of near surface layers of engineering materials. The test methods examined are: micro-tensile testing, bulge testing, ultra-low load indentation testing, and micro-fabricated test structures. The applicability of these techniques as well as their advantages and difficulties are examined. Special emphasis is given to recent developments in ultra-low load indentation testing and micro-fabricated test structures.

Introduction

The current high level of interest in the mechanical properties of near surface layers has been driven largely by three sources. The first driving force is the ongoing search for increased mechanical efficiency, a problem controlled by the wear and friction of contacting materials. The attempt to minimize both friction and wear of structural materials has led to the development of a variety of advanced methods to modify the mechanical properties of material surfaces. One of the most effective and promising techniques is ion implantation. A specific example of this technology which is now seeing engineering application is the implantation of nitrogen or carbon and titanium into steels (1-3). Such implantation layers are commonly only a few hundred nanometers thick and cannot usually be

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separated from the underlying material, presenting a difficult problem as to how to perform mechanical tests to determine their properties.

In the second case, the application of thin films and ion implantation to the fabrication of microelectronic devices has brought with it another unique set of testing difficulties. In large scale integrated circuits, implanted layers again may extend only 100 nm or so into the surface of the silicon substrate. Sputtered and evaporated metal films approximately 1 μm thick are deposited onto the surface of these devices to provide electrical interconnects. The successful development of solutions to problems such as stress voiding of interconnect lines (4,5) will require careful mechanical characterization of the metal lines as well as the semiconducting and dielectric layers which make up an LSI device.

The third case is the very recent development of micromachining technology for sensors and actuators (6-9). This fast growing technology has introduced the need for test methods to measure the mechanical properties of micromechanical elements lithographically fabricated from silicon. Such measurements are necessary in order to set limits on the mechanical loads which can be expected of these extremely promising small machines.

The mechanical testing of relatively thick films or layers (say, >5 μm) is perhaps only slightly more difficult than testing of bulk materials. Standard techniques such as tensile testing and microhardness testing can be readily applied to such materials. For films or layers thinner than this, however, testing is much more complex. The possibility of mechanical damage during handling of very thin films and/or very small specimens which have been liberated from their substrate becomes a concern. Sufficient

sensitivity in the measurement of loads and displacements is difficult to achieve. This problem is particularly acute in indentation testing, where insufficient resolution makes it impossible to extract film properties from those of the combined film/substrate system. In general, complex measures must be taken to overcome these difficulties.

Very recently, a detailed bibliographic review of the mechanical testing of thin films and near surface layers has appeared (10). Rather than simply duplicate the bulk of this comprehensive work, the present paper will focus on a discussion of the relative merits and difficulties of the most popular test methods for thin films and ion implanted surfaces and will provide an introduction to new techniques which show promise to advance the state of micromechanical testing. Ultra-low load indentation techniques and micro-fabricated test structures will receive special emphasis due to recent developments in the application of both techniques. These developments promise to extend the utility of these techniques to specimens which previously could not be successfully tested, providing a still wider choice of tools to the experimenter.

Discussion

Micro-tensile testing

Micro-tensile testing should probably be considered the most mature test method considered here (11-14). Conceptually, it is simply a small-scale version of everyday tensile testing used for the examination of bulk material specimens. However, because of the scale of specimen examined, unique problems are introduced into what is, in concept, a very simple test. In a general sense, several of these problems apply to other test techniques which will be discussed, so they will be treated in some

detail here.

Specimen fabrication and mounting must be performed with care in order to obtain meaningful test results. This is obviously true for testing at any scale, but is particularly difficult to achieve for the small, delicate samples we are concerned with here. Separation of the test specimen from its underlying substrate is the first problem which must be overcome. Commonly, thin film specimens are deposited onto cleaved rocksalt crystals (12,15) or mica (16) and then removed. Mounting of the specimen in the load frame must then be addressed. Gripping of specimens has been accomplished by gluing (11,12,15), friction (13,14,16), or, for extremely delicate samples, van der Waals forces (17). The technique used must allow the application of sufficient force to deform the specimen gage section, but must not induce enough damage into the grip end so as to precipitate a failure outside the gage section. Finally, tensile deformation is imposed on the specimen, either by means of a "conventional" mechanical drive system (13-15) or electromagnetically (11,12,16,17). The choice of loading technique can influence the test results: one example is yield phenomena such as load drops, which are not easily captured using a load control test (typically the case for electromagnetic systems).

At first glance, micro-tensile testing holds the potential to provide a true measure of the mechanical properties of a near-surface layer. Unfortunately, several difficulties exist which have kept the technique from realizing this apparent potential. First, and perhaps the most important, the technique is fundamentally limited to thin films which can be removed from their underlying substrate. In order to fulfill this criterion, it is frequently necessary to deposit the film to be tested on a substrate other than the one

most of interest. This can influence the resulting microstructure, and hence the mechanical properties. Also, because of this constraint, near-surface layers modified by ion implantation or some related technique which cannot be separated from the underlying substrate are clearly not amenable to micro-tensile testing.

In most applications, one is interested in the response of a film while it is attached to a substrate. Removal of the film will relieve any residual stresses the film may contain due to mismatch with the substrate. The resulting strength of the now-unstressed film will be different than one would measure for the film in the attached state.

An additional problem which can arise due to residual stresses is that the film may curl when removed from the substrate. Such distortion makes a film more difficult to handle, much more difficult to mount, and will obscure measurement of small strain tensile behavior. As the film straightens, the load-elongation record of the test will reflect the reduced load required to straighten the film instead of stretching it. Thus the test results will be colored until the film is fully straightened. As small strain behavior is the response best examined with micro-tensile testing, this can be a particularly vexing problem.

Another difficulty is that the technique generally requires thin sheet-type specimens whose aspect ratios of width:thickness and length:thickness are very large. Such a geometry leads to a test specimen which is prone to material and/or geometric instabilities and does not provide a good measure of the inherent ductility of the near-surface material. Thin sheet tensile specimens cannot deform very far under pure uniaxial strain conditions - rather the specimen localizes deformation into a very narrow

region inclined to the tensile axis and deforms to failure under plane-strain conditions.

Also, because the specimens are small and/or of very large aspect ratio, they tend to be prone to damage during handling prior to the test. For a ductile specimen, this damage generally takes the form of plastic bending, introducing work into the specimen which will obscure the actual small strain behavior of the thin film. For high strength or brittle specimens, shear banding or cracking may result, degrading the subsequent ductility and/or strength of the sample.

Additionally, both edge preparation (14) and axial alignment (13) of micro-tensile test specimens can strongly influence the test results. Poor edge preparation will typically leave geometric defects in the sample, which can lead to cracking or tearing. Misalignment introduces a buckling mode into the deformation of the test specimen, resulting in preferential straining along one edge of the sample, obscuring the true stress-strain response of the entire gage section.

Bulge testing

Bulge testing of thin films dates from the same time period as does micro-tensile testing (18), but the technique has seen less use. As the test is conventionally performed (18-22), bulge test films are first deposited on a substrate from which they can be easily removed. Next they are removed from that substrate, mounted in a test fixture and pressurized to failure. By monitoring the applied pressure and the displacement of the bulged film, one can obtain a fairly good measure of the biaxial stress-strain response of the material.

One possible advantage of this technique over micro-tensile testing is

that the potential for handling-induced damage is slightly smaller, as higher loads must be applied to deform the relatively wide, approximately square bulge test specimen as opposed to the narrow gage section of a micro-tensile specimen. Also, there is no need to be concerned over alignment of the sample, and specimen edge conditions do not influence test results, since the edge is outside the central section of the specimen which is stressed.

A drawback to bulge testing is that the test data necessary to calculate fundamental mechanical properties is not easily measured. This is because strain measurements are not easily made for bulge tests of thin films. The basic difficulty is that any form of contacting displacement probe tends to influence the displacement it attempts to measure. Also, the probe may damage the specimen, causing it to fail prematurely. Optical methods of displacement measurement such as laser interferometry (20) may be used in order to avoid this problem.

Like micro-tensile testing, bulge testing requires that the specimen to be tested be removed from the underlying substrate. However, assuming that the experimental system used can accomodate such a specimen, the near surface layer to be examined need not necessarily be completely separated from the backing material (23). Instead, it is sufficient that the substrate be etched or milled away from the back to provide a circular film specimen still bonded to the substrate around its circumference. A sample prepared in this manner may be less susceptible to handling-induced damage and may allow the preparation of bulge test specimens from extremely thin films.

Like micro-tensile testing, one of the biggest problems associated with

bulge testing is that the large film area sampled makes the specimens very prone to strain localization due to local perturbations in film thickness or properties. Thus, although it is an extremely useful technique to obtain the average biaxial small strain response of a thin film, it is somewhat limited for studies of gross plastic flow.

Ultra-low load indentation

Ultra-low load indentation is at present the most commonly used test method for examining the mechanical behavior of thin films and ion implanted surfaces. The deformation response of near-surface layers most often of interest to the experimenter is straining due to the application of very localized forces. As an example, this sort of localized deformation is generally present during sliding friction - plastic flow due to contact of microscopic surface asperities. Such deformation is not very well approximated by the tensile deformation of a large aspect ratio, liberated thin film. Thus, the very local deformation produced in ultra-low load indentation testing can have an advantage over the more global deformation field produced by micro-tensile or bulge testing.

Ultra-low load indentation tests are relatively simple to perform and can probe extremely thin surface layers (if a state-of-the-art test system is used). Additionally, this test technique provides a very useful tool for examining at the response of a near surface layer to very large deformations. Because the indent formed can be extremely small (perhaps 100 nm or so), ultra-low load indentation can serve as a non-destructive screening tool and can be used to look at the influence of sequential modification processes on the mechanical response of a near-surface layer. The material to be tested need not (and, indeed, in most cases should not) be removed from the

underlying substrate, and thus the material should not be damaged during handling. This also makes ultra-low load indentation extremely attractive for studies of ion implanted surface layers. Also, any residual stresses in the near-surface material will not be affected, and thus their influence on the mechanical response of the specimen will be examined by the test.

In its basic form, ultra-low load indentation testing suffers from the inherent problem that "hardness" is not considered to be a fundamental mechanical property. Rather it is simply a convenient measure of mechanical integrity. At best, it provides some indication of flow stress which may be useful for comparison with other materials or processes. Analytical models of the indentation test have been able to provide fair correlation between hardness and the compressive flow stress of the material being indented. This correlation, however, is not perfect and indeed ultra-low load indentation testing alone cannot hope to measure the complete yield behavior of a material. These problems have served to limit the applications of indentation testing at any scale for the determination of the mechanical properties of materials. Only recently have techniques been developed which promise to overcome some of these difficulties.

Ultra-low load indentation testing as practiced today is largely a result of the work of Pethica and co-workers (24-27). This group worked to develop an extremely sensitive test instrument and to properly characterize the influence of test parameters and indenter geometry on indentation test response. They demonstrated the validity of ultra-low load indentation as a tool to measure of the mechanical response of a near-surface layer. Development of the technique has continued (28-30), and useful methods now exist to calculate additional information about a material from the

indentation test. A number of other workers have also played a role in developing highly sensitive test instruments and applying them to the study of material systems not amenable to examination by other methods (31-39).

Indentation tests are not readily interpreted to provide measures of fundamental mechanical properties. Both simple analytical techniques (40,41) and complex numerical simulations (42,43) have been used in the attempt to convert hardness values to plastic deformation behavior; neither has proven to be completely successful. Approaching the problem from the opposite direction, Bourcier et al. (44) showed that indentation load-depth results can be analyzed using mechanical properties of the material being indented obtained from other mechanical tests. Specifically, it was shown that the load-depth history of indentation tests performed over a wide range of size scales and on a variety of materials could be successfully modeled using uniaxial compression test results as input for a large strain finite model of a specimen indented by an elastic indenter. Recently, we have applied this combined experimental/numerical approach to the study of the mechanical response of high purity aluminum implanted with oxygen ions (45). Fully annealed aluminum was implanted to a depth of approximately 500 nm with oxygen levels of 5, 10 and 20 at. %, followed by annealing at 450 and 550°C. Indentation load-depth results obtained using an ultra-low load test system developed at our laboratory (31) revealed large differences in the responses of the unimplanted and implanted material. However, the resolution of the test system used did not allow us to measure the response of solely the implanted layer - rather we were sampling the composite response of both the implanted layer and the underlying substrate. In order to determine more clearly the mechanical response of the implanted layer,

a large strain finite element model of the implanted specimen was constructed. The stress-strain response of the substrate was measured using uniaxial compression testing of annealed samples of the aluminum substrate. Properties of the implanted material in the model were systematically varied until the resulting predicted load-depth response agreed with the experimental results. The strengths calculated in this manner for the implanted material were in good qualitative agreement with calculations based on conventional theories of particle and coherency strengthening in the implanted layer. Although this technique shows great promise for the characterization of materials which do not readily lend themselves to any other test method, it is by no means a cure-all. The stress-strain response obtained by this technique (in its current state of development) is not unique. Indeed, what this method actually measures is the flow stress of the implanted layer at some average value of strain characteristic of the indentation test. Such a flow stress could in theory result fromany one of an infinite number of combinations of yield strength and plastic stress-strain response. Determination of detailed information about the stress-strain history of a modified surface layer requires the use of some other technique.

Micro-fabricated test structures

Progress has been made in the past few years in the development of test methods/specimens for thin films which circumvent the difficulties of those test techniques discussed above. Basically all of these approaches involve the fabrication of suspended beam-like microstructures which are either a) "self-testing" due to residual stresses (46-51) or gravity (52) or b) are mechanically deformed using an ultra-low load indentation tester

(53,54). Several of these new designs will now be discussed and compared with earlier techniques.

Simple beams

The basic technique of measuring the mechanical properties of thin film using cantilever beam specimens is now some ten years old. The first work (55) involved inducing resonant frequencies in the beam through the use of an oscillator attached to the substrate. Recently, ultra-low load indentation test machines (56,57) have been used to apply mechanical displacements to the cantilever beams. A very simple approach to the deformation testing of near surface layers has recently been developed at Stanford (53,54). It involves the fabrication via silicon micromachining techniques of cantilever microbeams of thin films which have been deposited on a silicon wafer. The beams are deflected using a commercially available ultra-low load indentation test instrument. The technique is most directly applicable to the determination of the elastic response of the test specimen, but also may be used to approximately measure yield strength. However, large strain plastic deformation cannot be measured using this cantilever beam technique, since interpretation of the very localized bending which occurs in the plastic hinge of the specimen is not currently possible.

A new micro-fabricated test specimen design

Very recently, we have started development of a different beam-like specimen design at our laboratory (58). The proposed test specimen is shown in Figure 1. It is a free standing doubly-supported beam with two reduced sections which serve as tensile specimens. The wide center section of the beam serves as the point of contact for a specially fabricated diamond test probe which displaces the sample into the etched cavity

beneath it. Conceptually similar doubly-supported beams have previously been fabricated of silicon (59-61). It is suggested that the specimen proposed here will be etched from aluminum alloy thin films which have been deposited on oxidized silicon wafers. Other fabrication methods might be employed to examine different film/substrate combinations.

As opposed to singly supported cantilever beams, which are best used to measure the elastic response of a surface layer, it is felt that this specimen will best be used to measure the plastic deformation of thin films. Large strain finite element analysis has been applied in the design of this specimen. A deformed mesh from one such simulation is shown in Figure 2. The finite element calculations predict that this sample will basically bend at the ends of both gage sections and will simultaneously stretch within the reduced gage sections on either side of the diamond probe. The forces required to bend the beam are quite small compared to the forces required to stretch the specimen gage sections. Thus the resulting force-displacement history predicted by the analysis is very strongly related to the tensile stress-strain response of the thin film. The analytical results display good resolution of the yield strength of the material and are sensitive to the rate of plastic strain hardening of the film. Through the combined use of experimental testing and numerical modeling of this specimen we will be able to accurately determine the tensile stress-strain response of thin film metallizations. Also, the specimen may be applicable to creep and stress relaxation testing in load and displacement control, respectively.

Conclusions

A flexible set of tools exist today to examine the mechanical properties of near surface layers of engineering materials. These test methods vary

widely in the specifics of their execution and their resulting strengths and weaknesses. Micro-tensile testing and bulge testing can provide fine characterization of the global small strain response of films, but the test specimens are prone to damage and the techniques are not applicable to implanted layers of bulk material. Ultra-low load indentation testing can examine the large strain response of near-surface layers which remain attached to their substrates, but analysis of the resulting load-depth data is extremely difficult. Micro-fabricated test structures hold the future promise of providing a wide range of mechanical properties characterization, but as yet have not been fully enough developed to be used on a routine basis. No one method is available at present which can probe the full range of mechanical response for near-surface layers which may be of interest. Rather,investigators must determine which test method(s) best satisfy the goals of their particular investigations.

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Figure captions

FIG. 1.--Proposed micro-fabricated test structure currently under study.

FIG. 2.--Finite element mesh showing predicted deformation of our proposed micro-fabricated test structure.

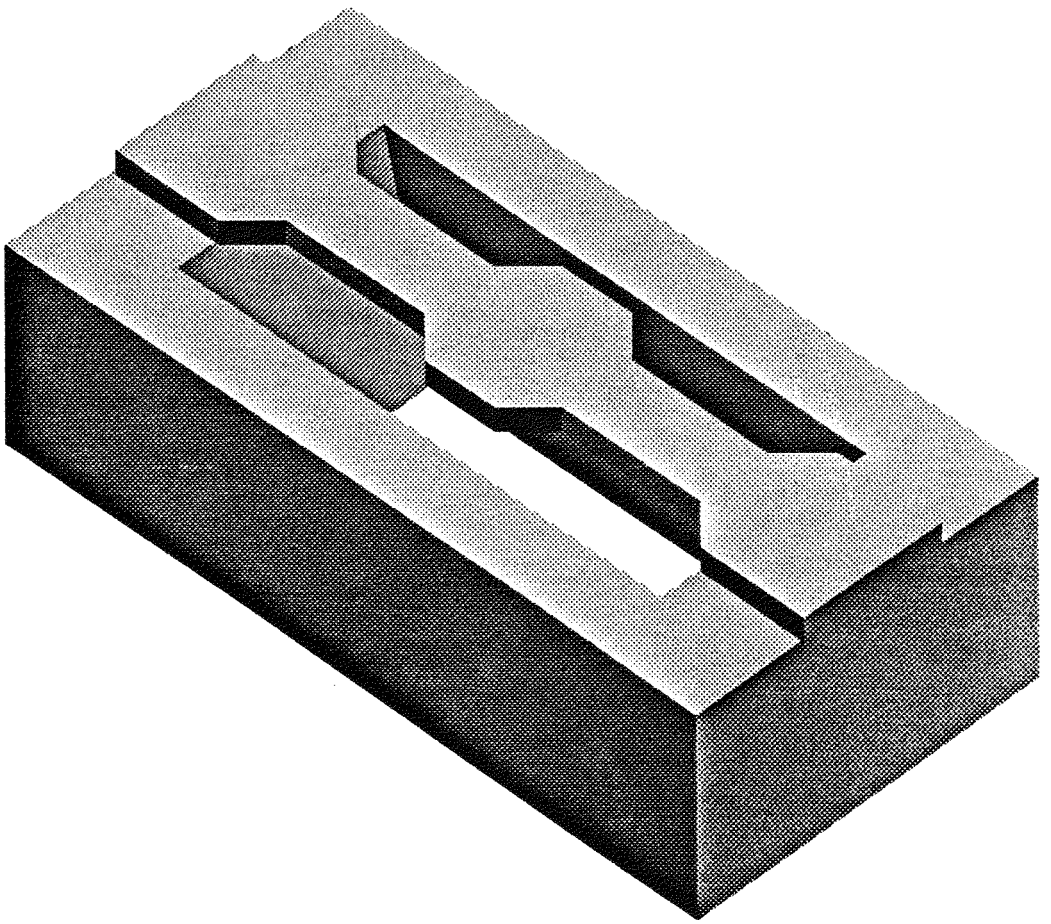

Fig. 1

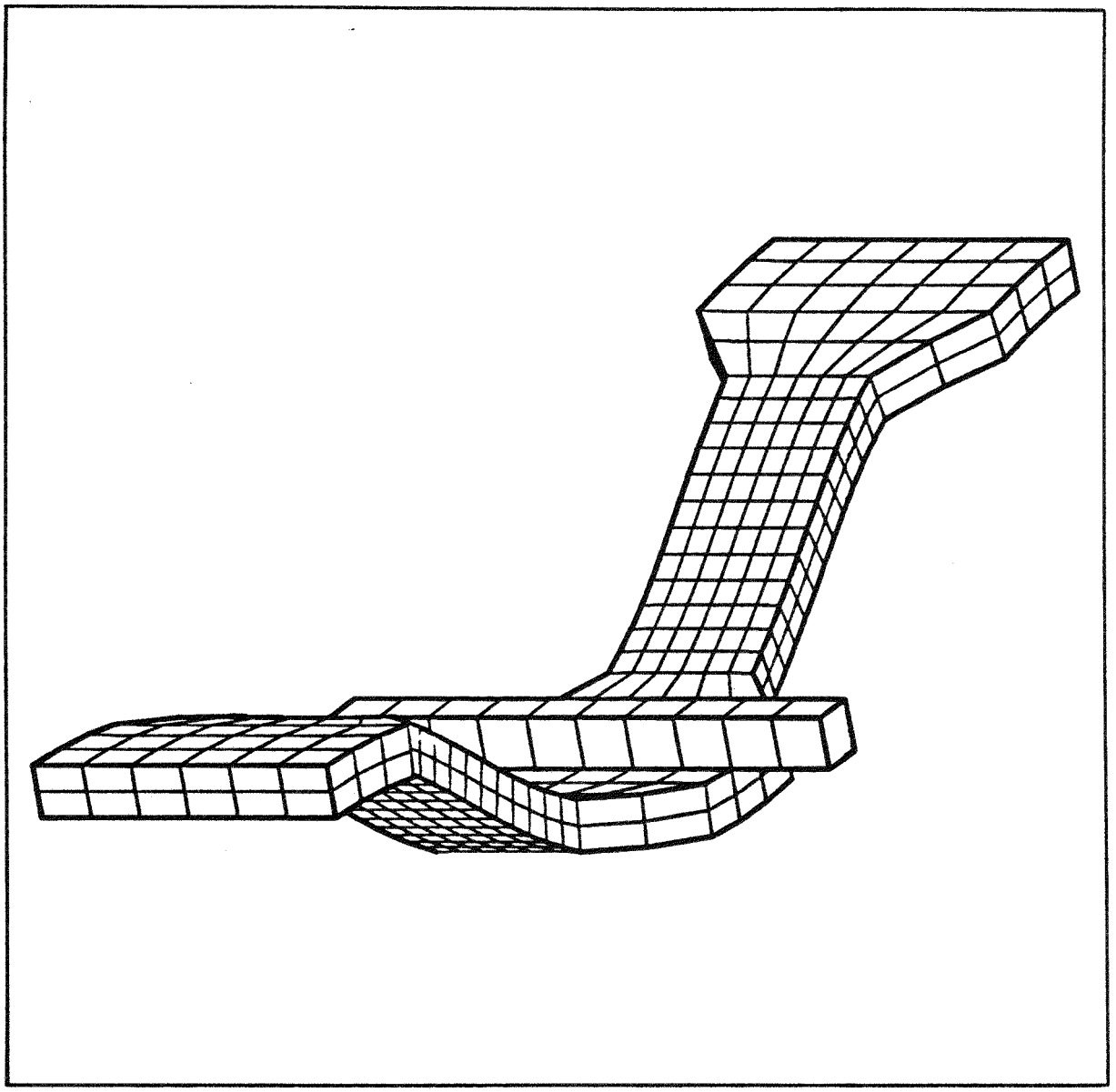

Fig. 2