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An investigation of the shape and dimensions of some diamond styli

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Abstract An appraisal is given of the qualities of diamond styli which are used for surface texture measurement. Some of the difficulties encountered in normal optical and electron optical methods of assessment are discussed, and a possible solution which involves a two-stage replica process is described.

1 Introduction

The assessment of surfaces by tactile trace instruments has found a great deal of practical use in industry because of the ease of interpretation of profile graphs as well as the convenience of having an electrical signal available for the quantitative analysis of various parameters. The increasingly stringent demand for a better understanding of the relationships between surface conditions and the functional characteristics of component parts has been greatly aided by the developments during the last decade in electronics, which have served to make possible the design of more sophisticated instruments. We are now at a point in the development of profilometric measurement of ultra-fine surfaces where the dimensions in the contact region of the probe are of the utmost significance. The chart record which is revealed by the tactile tracer instrument can differ slightly from the true profile for a number of reasons, for instance the loading of the stylus can sometimes deform materials with low moduli of elasticity and plasticity. Another source of uncertainty is the lack of knowledge about the geometry of the stylus tip. For the normal run of engineering surfaces down to some 0·1 μm CLA, these effects have seemed negligible, but below this they become of increasing significance.

This paper describes more accurately the finite dimensions of the stylus tip. The aims in this endeavour have been twofold: firstly to perfect some techniques for the rapid assessment of the stylus tip and secondly to make use of these techniques in an attempt to set some kind of acceptance limit to which stylus tips can be worked by traditional methods.

The tips considered were (i) the nominal 2·5 μm Talysurf tip, and (ii) the ultra-sharp nominal 0·1 μm Talystep tip.

2 Methods of measurement of styli

2.1 *Traditional*

For the measurement of small geometrical forms using the light microscope it is often difficult to discriminate between real geometry and diffraction phenomena when objects have dimensions which are close to the theoretical limit of resolution. The method of light microscopy used for any specific application may reveal certain aspects of the object by phase or path differences. However, it is usually found that the more sophisticated forms of microscope will detract somewhat from the maximum possible resolution for a given wavelength. At each lens interface a certain amount of diffusion is produced by reflection, surface asperities and, in some cases, dust, so that the number of optical components should be kept to a minimum. For the particular problem of diamond tip assessment it has been our experience that those techniques which are contrived to increase the contrast of the image do so at the cost of resolution.

No matter which configuration of microscope is used for examining styli, it is important that the objective lens be of the best quality with a large numerical aperture. For establishing the quality of an objective lens we use the star test, in which a minute point of light is viewed against a dark background. A suitable test object is an evaporated film on a glass flat containing small pinholes.

Examination of one of the smaller points of light at each side of focus will reveal any spherical aberrations present in the objective lens. It is only when all the rays of light are focusing to a near point that the appearance of the diffraction effect on both sides of focus will be identical. The method of illumination for this test is not critical, apart from the need for a high intensity light source, because a small aperture in these circumstances can be considered as a self-luminous object. Our experience has been that an objective lens must perform well according to this test otherwise it cannot be exploited.

From an examination of the micrographs in figure 1, it is evident that bright field (*a*) does indeed show the major boundary best, while phase contrast (*b*) is overwhelmed by the relatively large dimensions of the pyramid faces. The Normarski interference method (*c*) (Lang 1968, 1969), whilst detracting slightly from the resolution of the boundaries, does show something of the texture on the tip itself. Figure 1(*d*) shows a chisel tip stylus as is used on the Talystep instrument for the measurement of very fine surface texture. This micrograph appears to indicate that the sharp edge of the chisel is about 0·2 μm across, but figure 3(*b*) reveals that it is less than 0·1 μm.

The extraction of reliable information from objects whose dimensions are close to the theoretical limits of the light microscope is difficult. When a craftsman is using a light microscope to monitor the various stages in the operations of diamond polishing, there is a practical limit of about 0·2 μm beyond which he may only proceed and estimate the degree of his success by guesswork.

2.2 *The scanning electron microscope*

For the analysis of the geometrical form of solid specimens the technique of electron bombardment of the object in order to generate secondary electron emission will have two main advantages over light microscopy: first, a large increase in the depth of focus is made possible by long-focus magnetic lenses which will keep the beam divergence small; second, an increase in the useful magnification will be attainable provided that the object is not too greatly affected by the electron beam, for example the object may acquire a charge or even suffer a change of its chemical composition.

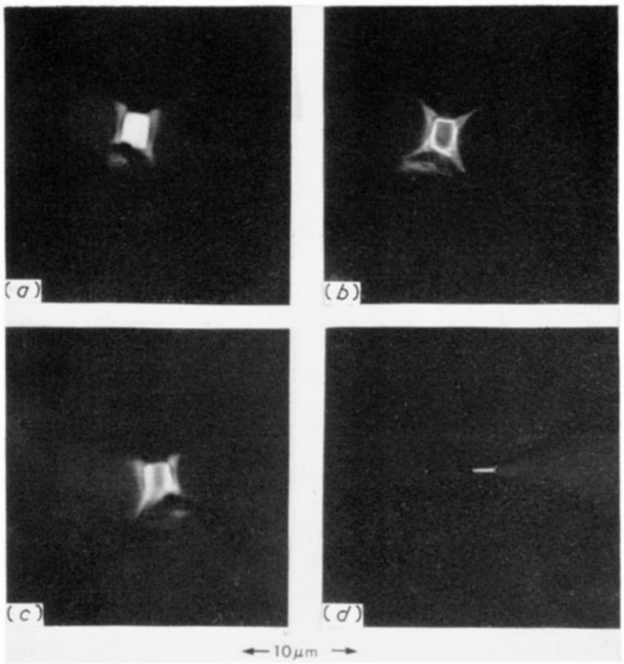

Figure 1 (*a*) Bright field, (*b*) phase contrast and (*c*) Normanski interference. For purposes of comparison, reference should be made to the electron micrograph (*a*) in figure 3 which is this same tip at a magnification of 9000×. (*d*) Chisel type stylus tip which can be compared with the electron micrograph (*b*) of this tip at 9000×. The objective lens used to obtain the micrographs in this figure was an exceptionally good 140X oil immersion of 1·3 numerical aperture

Among the variables which determine the resolution and contrast of the displayed image are shape, density, electrical conductivity and the secondary emission ratio of the various parts of the object, which is a function of the primary electron energy. Experimental data suggest that a primary electron at normal incidence to diamond and having an energy of 750 eV will release a maximum of about 2·8 secondary electrons. Ideally, what is wanted is a yield in energy which is unity with respect to the energy of a primary electron as this will minimize the tendency for potential gradients to develop. Secondary emission of an insulator such as diamond can be a complex phenomenon because of the absence of free electrons. An insulator will not lose energy as in a metal by interaction with free electrons in the conduction band. Primary electrons will only lose energy by interaction with valence electrons, and unless the insulating object is a thin film on an electrically conducting base, a space charge forms in the material. It is possible for an insulator to emit more electrons than were introduced, giving rise to a net loss of charge. The implications of this internal charging become evident when the geometry of the specimen is taken into account; charges will tend to collect or migrate towards peaks or other sharp boundaries.

The higher the energy of the primary (incident) electrons the further will they penetrate, and the number of electrons in the solid gaining energy from them will be greater. Electrons accelerated by 20 kV will penetrate solids such as aluminium to a depth of the order of 1 μm and it is expected that the penetration of electrons into diamond will be almost double this. There is evidence that surface contamination and thin conducting films used on specimens in scanning electron microscopes can greatly affect the emission properties; however, prevention of the charging effects in thick insulating objects would require an inordinate thickness of the conducting layers.

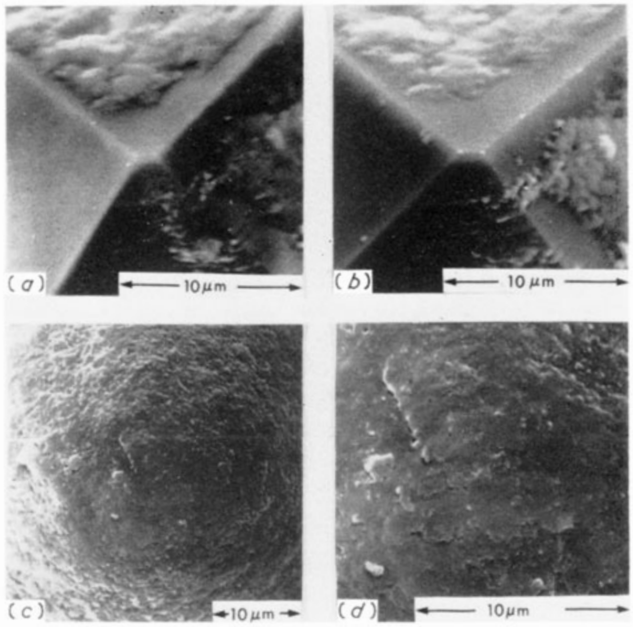

Figure 2 Electron micrographs from a stereoscan microscope: (*a*), (*b*) chisel tip stylus in the (*a*) uncoated and (*b*) the coated state. The coating is of aluminium and is about 20 nm thick; (*c*), (*d*) typical gramophone stylus whose tip radius is about 12 μm. The coating in this case is gold and about 10 nm thick

We find that overdeposited conductors such as aluminium, gold and carbon have little effect on the instrumental resolution at the diamond tip.

Figure 2(*a*) and (*b*) shows that the resolution at the extreme tip is somewhat increased by vacuum overcoating with 20 nm of aluminium; however, the gain is not sufficient for an accurate measurement of the sharp dimensions of the chisel to be made. The matter visible around the tip is dirt. We have sometimes found that an appreciable increase in resolution is evident in the near neighbourhood of such foreign substances.

The suitability of the scanning electron microscope for diamond tips on which there are no really prominent features is shown by plates (*c*) and (*d*), which illustrate a standard gramophone stylus having a tip radius of about 12 μm.

2.3 *The transmission electron microscope*

Some single-stage replicas were tried using the familiar materials, gelatine, collodion and Formvar, as well as some two-stage techniques which utilized wax, gelatine, acetate and metals as the first stage. Our lack of success with the various well-known techniques was for the most part due to the stringent requirement for a replica that not only showed the tip dimensions clearly but also a good deal of the pyramid leading to the tip. This sort of deep replica is easy to find in an area which may be littered with various shapes and markings.

Glass as a first-stage replica material was considered in view of the problems encountered with other materials. Examination of a range of glasses reveals that certain liquids become particularly viscous near their freezing point, thus preventing the formation and growth of crystal nuclei. If cooling of such a liquid is continued beyond a region where the mass becomes rigid, then the random atomic structure which is characteristic of glass will be held in the solid state. The amount of ordering of the structure of glass that is possible is strongly dependent upon the cooling rate, so that the final configuration of the glass will depend on its composition and thermal history.

The qualities of glasses are quite different from those of the organic polymers which are usually used for replicas in electron microscopy, these substances being partly crystalline and only to some degree amorphous. If a glass could be softened to flow around the tip of a diamond stylus, then a good replica might well be formed. The advantages of such a replica would be that an accurate representation of the tip should result because of the amorphous nature of glass and because of the ease of taking a second replica from the glass by the evaporated carbon technique.

As the hardness of glass (soda lime) is 530 on the Knoop scale, while diamond is placed at 7000 on this same scale, the relative hardness of diamond and glass will be about the same as that of tungsten carbide and zinc. Accordingly one might not expect damage to even a fine stylus tip when it is indented into glass, provided that no lateral shear forces can operate.

A small rig was constructed for use on a standard instrument (Talystep), which enabled a known load to be applied while the diamond was resting on the glass. The instrument could then be used to monitor the total amount of plastic deformation which occurred on application of load.

The criterion used to make good replicas is that the force is applied smoothly and within a short period of time. Having such control enables many such indentations to be made within a defined area. The replica is then shadowed and carbon is applied from at least two different directions to improve the rigidity of the replica. An example of how the replica can fracture on removal from the glass is shown in figure 3(*b*).

The stylus shown in figure 3(*a*) is the type of tip usually employed for surface texture measurement and is quite adequate for the assessment of surfaces produced by most machine processes used nowadays in industry. Figure 3(*b*) shows a stylus tip which is necessary to resolve the very fine texture associated with polished or lapped surfaces having a directional finish. In figure 3(*c*) and (*d*), a stylus of exceedingly small tip dimensions is shown. With such a tip it is possible to measure very fine randomly orientated surface asperities such as are found on evaporated films and in certain conditions of devitrification which take place during annealing processes, given the proviso that only very low stylus forces can be used.

3 Discussion

Use of the more precise information about styli obtained by the methods described has already made possible a better understanding of some of the smaller features on surfaces (Whitehouse and Archard 1970).

We now know with a fair degree of certainty, just how far the spatial resolving power of the tactile tracer instrument can be effectively taken. Under normal loading conditions this limit will approach 0·1 μm or about $\frac{1}{4}$ of the wavelength of ultraviolet light. Further information can be obtained by virtue of the fact that if the stylus dimensions are known then they can be partly computed out of a profile chart. The argument does however have limitations in that on the one hand the positive asperities can have the stylus error removed but negative valleys into which the stylus cannot fully penetrate must be computed, although such computation can be made reasonably accurately.

The effects of two different stylus tip dimensions can be seen in figure 4 where chart (*a*) shows the profile of a small section of gauge block as measured by the relatively large tip (figure 3(*a*)), and chart (*b*) is a portion of this same surface as measured by the fine chisel tip stylus (figure 3(*b*)). Comparison of the charts (*a*) and (*b*) clearly shows that the

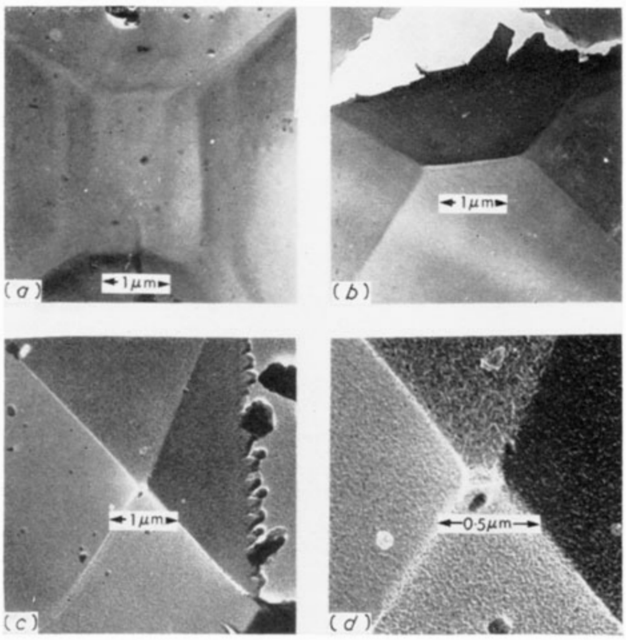

Figure 3 (*a*) Typical tip as used on our Talysurf instruments; (*b*) Talystep sharp stylus which is used for resolving very fine surface texture; (*c*) super-sharp stylus tip that was made in our laboratory; (*d*) an enlarged view of this very sharp tip

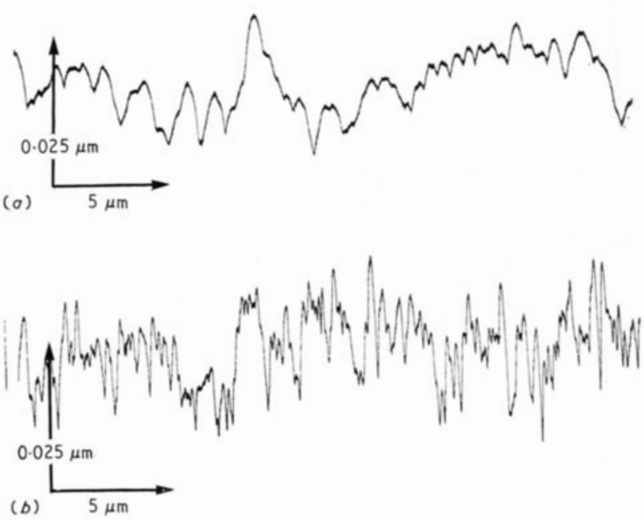

Figure 4 (*a*) Profile of a small section on a finely lapped gauge block using the stylus shown in figure 3(*a*); (*b*) a section of this same surface as measured by the stylus shown in figure 3(*b*)

larger stylus tip rides over and loses fine detail. A knowledge of tip dimensions is desirable in microhardness testing, where within the range of 100 g to 1 mg loads, indents are made into specific inclusions within polycrystalline bodies.

In ultramicrotomy where diamond knives are used to section specimens for electron microscopy, this method might play a part in defining the quality of the diamond knives used and their rate of wear.

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