Test Probe for Materials Testing Apparatus

This invention relates to a test probe suitable for use in materials testing apparatus and to apparatus incorporating such a test probe.

Indentation and scratch testing of materials is generally performed using a relatively hard test probe that is either impressed into a surface or drawn across a surface under constant or variable loads. Test probes usually include a tip made from diamond or ceramic crystals that have been ground and polished to a well-defined geometry, and a holder on which the tip is carried. The holder is so designed that it can be connected to testing apparatus for use, for example, apparatus disclosed in EP-A-1 095 254 of the present applicant.

For testing at room temperature or moderate temperatures, the crystal can be safely bonded to a holder by means of a polymer adhesive or a metal braze. Polymers are generally unsuitable above about 100°C, or in some chemically aggressive atmospheres, because in such conditions, they tend to soften or eventually decompose. Brazes can be safely used to higher temperatures, typically up to about 400°C, but various brazing constituents tend to react with atmospheric gases or to evaporate as the temperature is raised further. Evaporation causes contamination of the tip of the test probe and of a test sample that is placed adjacent to the tip of the probe. Therefore, evaporation is problematical both in air and in a vacuum.

An aim of this invention is to provide a test probe that can be used at elevated temperatures without suffering from the disadvantages of conventional test probes.

To this end, from a first aspect, this invention provides a test probe for use in materials testing apparatus comprising a probe holder adapted to be mounted for use on materials testing apparatus and a probe tip carried on the probe holder, in which the probe tip is secured to the probe holder by a body of ceramic cement.

Since the present invention provides a ceramic bond between the tip of the test probe and its holder, it can be safely used significantly beyond the temperature range available to conventional probes, both in air or in vacuum, without experiencing material degradation.

The probe tip is typically formed from a piece of hard, crystalline material, such as diamond or a crystalline ceramic. Alternative materials, suited to particular situations, may be chosen, such as sapphire or cubic boron nitride. The probe tip is most typically ground and polished to a predetermined shape and dimension.

The cement is preferably of a type that is cured chemically rather than by evaporation. It is chosen to have refractory properties. The cement may include a soluble silicate and more specifically may comprise a cured mixture of sodium silicate or potassium silicate and zirconium silicate.

The probe tip may be mounted on a flat surface of the probe holder or within a recess formed within the probe holder. Alternatively, the probe tip may be in a space within the probe holder and arranged to project from the probe holder. In such embodiments, the cement occupies a volume surrounding the probe tip within the space. Embodiments of this type permit mechanical location of the tip within the tip holder to supplement the action of the cement.

The probe holder may be of stainless steel; other metals such as molybdenum; alloys such as austenitic nickel-chromium-based superalloys, e.g., Inconel (rtm); or of refractory material.

From a second aspect, this invention provides a method of making a test probe embodying the first aspect of the invention. The method may involve applying cement in an uncured state to the probe holder, pressing a crystal into the cement. Alternatively, the method may involve introducing a crystal into a space within the probe holder such that it partially projects from the probe holder, introducing uncured cement into the space, causing the cement to surround the crystal within the probe holder. In either case, the method may further involve curing the cement, and forming the probe tip to the required shape and dimensions. When forming the probe tip to the required shape and dimensions, both the crystal and the cement may be subject to an operation involving one or more of grinding and polishing. Full hardening of the cement is achieved, typically as a final step, by heating the probe to a temperature close to the maximum operating temperature of the probe and in an appropriate atmosphere, for instance to a temperature of approximately 750°C in the preferred embodiment.

From a third aspect, this invention provides materials testing apparatus incorporating a test probe embodying the first aspect of the invention.

From a fourth aspect, this invention provides a method of testing materials comprising the steps of: mounting a sample of the material in apparatus embodying the second aspect of the invention; and causing the test probe to make impact or abrasive contact with the sample.

In a method embodying this aspect of the invention, the probe and/or the sample may be heated. The temperature to which it or they are heated may be in excess of 100°C and may be in excess of 500°C.

Embodiments of the invention will now be described in detail, by way of example, with reference to the accompanying drawings, in which:

Figures 1 and 2 are cross sections through test probes being first and second embodiments of the invention;

Figure 3 shows a plunger tool used in manufacture of the embodiment of Figure 6;

Figure 4 shows, in cross section, a first step in the manufacture of the probe of Figure 6;

Figure 5 shows, in cross section, a second step in the manufacture of the probe of Figure 6; and

Figure 6 shows, in cross section, a third embodiment of the invention.

With reference to Figures 1 and 2, a test probe embodying the invention comprises a tip 10 carried on a probe holder 12 to which it is fixed by cement 14. In these embodiments, the tip 10 is retained in place solely by the cement 14.

In the embodiment of Figure 1, the probe holder 12, which is machined from a block of stainless steel, comprises a cylindrical body part 20 and a head part 22 that is shaped as a truncated cone, coaxial with the cylindrical body part 20, with a flat mounting surface 24, which is generally normal to the axis of the cylindrical body part 20.

The embodiment of Figure 2 is similar to that of Figure 1, except that the head part 26 extends further from the cylindrical body part 20. An axial blind bore 28 is formed in the head part, open facing away from the cylindrical body part 20. This provides additional mechanical location of the tip 10. Retention of the tip 10 can be further enhanced by undercutting the bore 28, either to form a dovetail shape or with a radial groove, as shown in dashed lines in Figure 2. In such an embodiment, the cement cannot fall out of the recess once it has cured.

The probe holder may alternatively be made of a refractory material, but otherwise be similar to those described above.

In this embodiment, the cement 14 is a ceramic cement that is prepared by premixing 20% sodium silicate solution and 80% zirconium silicate powder by weight. The sodium silicate solution is an aqueous solution of specific gravity 1.37 g/cc (1370 kg m^{-3}) formed from a mixture of SiO_2 andNa_2O at a ratio of 3.22:1 by weight. The cement begins to set at room temperature within a few minutes of being mixed. In this example, the cement used is the High Temperature CC cement manufactured by Omega Engineering, Inc.

To assemble the test probe, cement is applied to the mounting surface 24 or into the blind bore 28, as the case may be, immediately after mixing. A crystal is pressed through the cement 14 until it makes contact with the underlying material of the probe holder 12. The cement is allowed to cure at room temperature for 48 hours. In this condition, part of the crystal may project from the cement, or it may be entirely encased within the cement.

The test probe can then be ground and polished such that the crystal and the surrounding cement 14 is formed into a tip 10 of the required shape and size.

Finally, the probe is heated (in this embodiment to 750°C) to achieve full hardening of the cement.

For ceramic cements with relatively large particulates or lower adhesive forces, it is advantageous to supplement the ceramic bonding with mechanical clamping resulting in a test probe as shown in Figure 6.

This embodiment comprises a tip 30, a probe holder 32 and a body of cement 34.

The probe holder 32 has a cylindrical body part from one end of which extends a head part 36 in the shape of a truncated cone. An axial bore 38 extends the length of the probe holder 32. An end part 40 of the bore 38 remote from the head part 36 is tapped with an internal thread. Within the head part 36, the bore 38 is tapered.

A plunger tool 50, as shown in Figure 3, is used to assist in construction of the test probe of Figure 6. The plunger tool has the general configuration of a bolt having a hexagonal head 52, and a shaft 54 that extends from the head 52. The shaft 54 has a threaded length close to the head, and remote from the head, an unthreaded length 56 of lesser diameter. The lengths of the shaft 54 are selected such that the threaded length can engage with the thread tapped in the bore 38 of the probe holder 32 and that the unthreaded length 56 is a close fit within the part of the bore 32 that is not tapped.

To assemble the test probe, a crystal 60 is first introduced into the bore 38 of the probe holder 32 through its opening remote from the head part 36. The crystal 60 is selected to be of a size that allows part of it to emerge from the bore where it opens at the head part 36, but such that it is too large to allow it to pass completely out of the bore 38, instead becoming trapped in the tapered part of the bore 38. A body of freshly mixed cement 34 is then introduced into the bore 32 through its opening remote from the head part 36.

The plunger tool 50 is then inserted into the bore 38, with the threads of the plunger tool 50 and the probe holder 32 in threaded interengagement. The plunger tool 50 is turned using its head 52 to drive it into the bore 38 of the probe holder 32. The unthreaded part of the shaft 54 of the plunger tool 50 acts as a piston within the cylinder formed by the unthreaded part of the bore 38, so driving the cement 34 towards the head part 36. There, the cement 34 is forced to surround the crystal 60 and fill the space surrounding the crystal 60 within the bore 38, as shown in Figure 5.

The cement 34 is then cured and the crystal 60 and head part 36 formed into a probe tip 30 as described above.

In these various embodiments, the crystal 60 is ground and polished using well-known techniques to form a probe tip from it. If the crystal is fully embedded in cement or placed within a holder that supplements cement with mechanical retention, the polishing includes an operation that also shapes and polished the cement, the material of the probe holder (or both) as necessary to form and expose the required probe tip geometry. This may be, for example, pyramidal, spherical or cylindrical

In use, a test probe embodying the invention may be heated through contact with a heated specimen, or by pre-test heating of the test probe assembly by means of an attached heater.

The coefficients of thermal expansion of the components of the test probe (including the probe holder and cement) are matched as far as possible to preclude differential expansion and consequential potential cracking of the cement.

The completed test probe can then be mounted within a test apparatus and used to perform tests on samples of material in a manner well-known to those skilled in the technical field. Such tests may be performed at elevated temperatures without the risk that the test probe will degenerate or damage the sample.