Thermal contraction of ultrahigh vacuum materials for scanning probe microscopy from 300 to 4 K

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The total thermal contraction of UHV materials commonly used in the construction of scanning tunneling and scanning force microscopes has been measured at 195, 77, and 4.2 K. The measurements were made with a simple push-rod dilatometer whose accuracy was checked against the known thermal contraction of 304 stainless steel. The results of the measurements indicate that the total thermal contractions of Macor machinable ceramic and the lead–zirconate–titanate piezoceramic PZT-5A are closely matched and similar to the contractions of titanium and tantalum. The total thermal contraction of Torr Seal UHV compatible epoxy is substantially larger and is similar to that of other filled epoxies. © *1995 American Vacuum Society.*

In the dozen years since its invention, the scanning tunneling microscope[1] (STM) has found wide application in the field of surface science as a probe of topography and electronic structure on an atomic scale. The success of the STM has also inspired a fast-growing variety of related techniques based on atomic (van der Waals) forces, magnetic forces, capacitance, and near-field optics, to name but a few.[2] The wide applicability of these scanned probes has led to a growing interest in microscopes which can operate at cryogenic temperatures, and a number of researchers have reported on UHV instruments which operate in the low-kelvin range.[3–5] In designing these cryogenic scanning probe microscopes, it is of course very important to know the low-temperature voltage response of the piezoelectric scanning element. A number of groups have reported on the low-temperature piezoelectric coefficients of the *de facto* standard tube-shaped scanners made from the lead–zirconate–titanate ceramic PZT-5A.[6–8]

In order to design a microscope to close tolerances, and to avoid the destruction of delicate components through differential thermal contraction, it is equally important to know the low-temperature thermal contraction of these piezo elements, as well as those of the other materials used in constructing the microscope. While researchers in the field of low-temperature physics have built up quite a store of knowledge about the thermal contraction of unusual materials,[9] only very limited information is available about the thermal contraction of the ceramics, piezoceramics, and adhesives commonly used in ultrahigh vacuum and scanning probe microscopy experiments. To date, only one low-temperature measurement of a PZT material (PZT-8) has been reported in the literature.[10] In this Brief Report, we present data on the total thermal contraction of piezoceramic tubes of the type commonly used as scanning probe microscope (SPM) scanners, as well as measurements of the low-temperature contraction of Macor machinable glass ceramic[11] and Torr Seal UHV compatible epoxy.[12]

Our measurements were made using a simple push-rod dilatometer similar to the design of Swift and Packard.[13] This device, illustrated in Fig. 1, compares the contraction of a 6 mm diameter by 20 cm long rod-shaped sample with the contraction of identically dimensioned rods made from oxygen-free high-conductivity (OFHC) copper. The test assembly is supported from a set of thin-walled stainless steel tubes, and any change in the dimension of the sample under test, relative to that of the copper, is transmitted to room temperature via a push rod (also a thin-walled stainless steel tube). To ensure that the support tubes and the push-rod contract identically, they were cut from the same stock material. The relative contraction or expansion of the test piece is read out by a machinist's dial indicator[14] with 1 μm resolution and 5 mm total range. Cooling of the dilatometer and samples was obtained by direct immersion in liquid baths at 195, 77, and 4.2 K.

Because the dilatometer measures only the difference in contraction between the reference rods and the sample under test, it is important to use a reference whose total thermal contraction has been reliably determined. If the length of the sample at a given temperature is denoted L_T, then the total thermal contraction is defined as $(L_T - L_{295})/L_{295}$, and is the sum of the observed differential contraction and the known thermal contraction of the reference. In the measurements reported here, OFHC copper was used as the reference material, because its thermal contraction is well documented and has been shown to vary only negligibly from sample to sample.[15,16]

We calibrated the dilatometer using test samples of OFHC copper and verified its operation with test samples of type 304 stainless steel. We observed an approximately 2 μm differential contraction using the copper test piece, which corresponds to a systematic error of $\sim$0.5%. When the data for the total thermal contraction of the stainless steel were corrected for this small effect, our results were found to agree with published values[17,18] to within $\sim$1%, as is shown in Fig. 2.

The results of our measurements on Macor, Torr Seal, and a PZT-5A tube are summarized in Fig. 3. The Macor sample (open circles) was cut to length from an ''as supplied'' 6 mm diameter rod. The Torr Seal sample (filled triangles) was

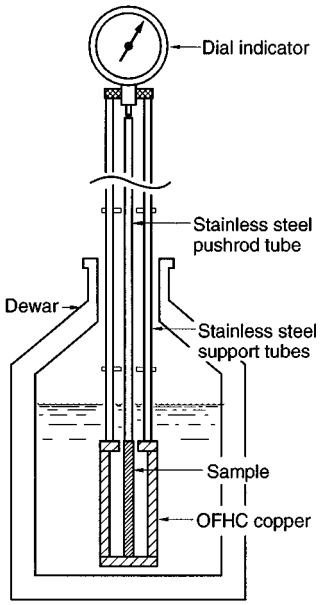

FIG. 1. Sketch of the dilatometer used in the measurements.

made by mixing the epoxy resin and hardener according to the manufacturers instructions, and injecting the mixture into a 6 mm diameter Teflon mold. After hardening, the rod of epoxy was extracted from the mold and cut to the correct length. Care was taken to use a sample of epoxy free of large air bubbles, which were found to make the rod bend on cooling. The dashed line in Fig. 3 is the thermal contraction curve for Araldite epoxy resin[17] scaled to match the total contraction of Torr Seal at 4.2 K. The total contraction of Torr Seal at 4.2 K is actually about half that of Araldite, which is a pure epoxy resin, but is quite similar to the contraction of epoxies which have added alumina powder to reduce their coefficient of expansion.[9] (Torr Seal consists of an epoxy resin with added talc and quartz powder.) Error bars for both the Torr Seal and the Macor are approximately given by the symbol size.

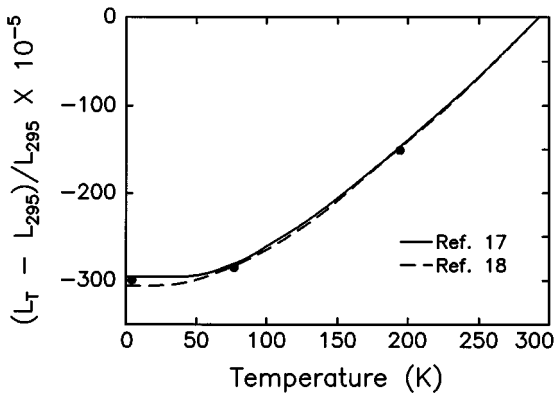

FIG. 2. Comparison of our measured values for the total thermal contraction of a sample of type 304 stainless steel at 195, 77, and 4.2 K (filled circles) with published values.

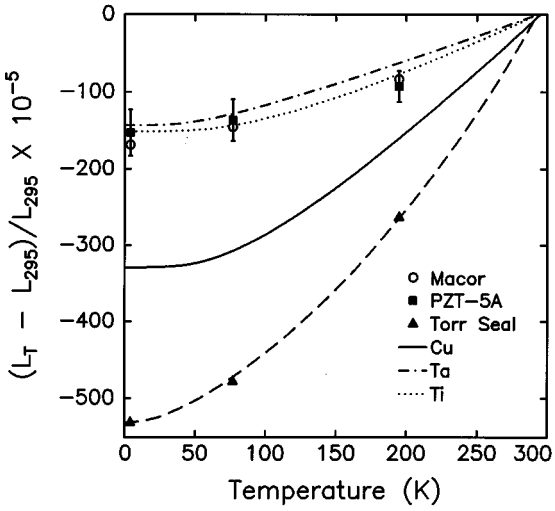

FIG. 3. Measured values for the total thermal contraction of Macor (open circles), PZT-5A (filled squares), and Torr Seal (filled triangles) at 195, 77, and 4.2 K. Error bars for the Macor and Torr Seal are approximately given by the symbol size. The error bars for the PZT-5A are considerably larger because that sample was only about 10% as long as the others. Also shown for comparison purposes are the total contractions of tantalum (dashed-dotted line), titanium (dotted line), and OFHC copper (solid line). The dashed curve represents data on the contraction of Araldite epoxy scaled to match the observed contraction of Torr Seal at 4.2 K. References are given in the text.

The PZT-5A tube used in these measurements had an outside diameter of 6 mm, a wall thickness of 0.5 mm, and unsegmented electroless nickel electrodes on the inner and outer surfaces. The material was poled in the radial direction. Our measurements therefore give the total thermal contraction perpendicular to the poling direction. To avoid problems associated with strain-induced electric fields in the sample, we shorted the inner and outer electrodes together with a dab of conducting epoxy. The manufacturer of this tube[19] was only able to supply us with a sample whose overall length was 2.4 cm. The absolute contraction of this tube was therefore considerably smaller than that for the longer samples, which is reflected in the larger error bars seen in Fig. 3. We find that the total thermal contraction of PZT-5A at 4.2 K is $150\pm30\times10^{-5}$, which is about 36% larger than the contraction reported for a slab-shaped sample of PZT-8 material.[10] The measurements reported in Ref. 10 indicate that PZT-8 actually *expands* on cooling along an axis parallel to the poling direction. While our apparatus was not designed to detect the increase in tube wall thickness that would accompany an analogous effect in PZT-5A, we would expect this change to be on the order of 0.5 μm and to be completely negligible in comparison to the reduction in tube diameter associated with contraction perpendicular to the poling direction.

It is interesting to note that the total thermal contractions of Macor and PZT-5A are extremely well matched, which suggests that Macor is an excellent material from which to fabricate scanning probe microscopes, from both thermomechanical and electrical points of view. For comparison, we have also included in Fig. 3 data on the total thermal con-

traction of titanium and tantalum,[17] two metals which are commonly used in UHV applications and which have total thermal contractions similar to Macor and to PZT-5A scanner tubes.

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