



AFM as an alternative for Young's modulus determination in ceramic materials in elastic deformation regime

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ABSTRACT

A novel alternative to the conventional nanoindentation technique for hard materials is presented. An atomic force microscopy probe is used as an indenter, applying loads in the nN range and producing elastic deformations of a few nanometres. This new technique allows a reduction of the different inherent nanoindenter problems. This result in an increase in the quality of the overall results, and thus provide better understanding of the contact mechanism between the indenter and the sample, yielding Young's modulus values consistent with the literature. This is confirmed by a series of experiments performed on different $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ textured and single crystal samples textured using different techniques.

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1. Introduction

It is well known that high- T_c superconductor's exhibit high critical current densities and high trapped magnetic fields at cryogenic (i.e., liquid nitrogen) temperatures. However, practical applications of these superconducting materials are often limited by their poor mechanical performance. Hence, mechanical properties such as hardness, microhardness, Young's modulus (E) and creep behaviour are crucial to the industrial applications of these materials. $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ or Y-123 ceramics have a useful combination of hardness, wear resistance, corrosion resistance, and toughness. This makes them excellent candidates for structural applications such as flywheels, cables and motors. Precise and quantitative knowledge of E values is a prerequisite for successful manufacturing and operation of these devices. However, very little information is available in the literature. Nowadays, it is widely accepted that a correct understanding and characterization of the E values during the working lifetime of textured $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ samples is critical to any desired structural application.

Picoindentation by means of atomic force microscopy–force spectroscopy (AFM–FS) has several advantages over standard methods with regard to the quantification of E values for $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ samples. First, the measurements are extremely local (small contact areas between the sample and the probe, of few nm^2) and can be

performed on different areas so as to average the mechanical properties of the sample. Second, it is a form of depth–sensing, so it allows characterization of a material at different penetration depths without surpassing the yield strength (σ_{ys}). Finally, it is not necessary to visualize and characterize the imprint produced during the indentation test, which simplifies the calculation of the E values. These advantages mean that picoindentation is a suitable and reliable technique for measuring E values while applying loads (F) ranging from a few nN down to the pN level. The study of force versus displacement (F – z) curves can shed new light on the elastic properties of hard materials, as these curves are similar to those obtained with classic macroscopic or nanoscopic indentation tests. The main differences involved in picoindentation are the reduced applied load and the nanometric contact area (A_c).

Fig. 1 is a schematic of the whole indentation process. Fig. 1a shows a model F – z curve (also known as force curve), which is the AFM data output after performing the picoindentation. When the AFM tip is far from the sample surface, there is no cantilever deflection (Δz), as there is no interaction between the tip and the surface (Fig. 1b). Then, as the AFM probe moves towards the sample, a process known as *jump to contact* takes place; the AFM probe bends downwards due to van der Waals and water meniscus interactions, which bring the probe into contact with the sample (Fig. 1c). As F increases, the cantilever deflection also increases and the sample is compressed (Fig. 1d). During the unloading process (that is, as the AFM probe move back to its initial position far from the sample) another process known as *jump off contact* occurs

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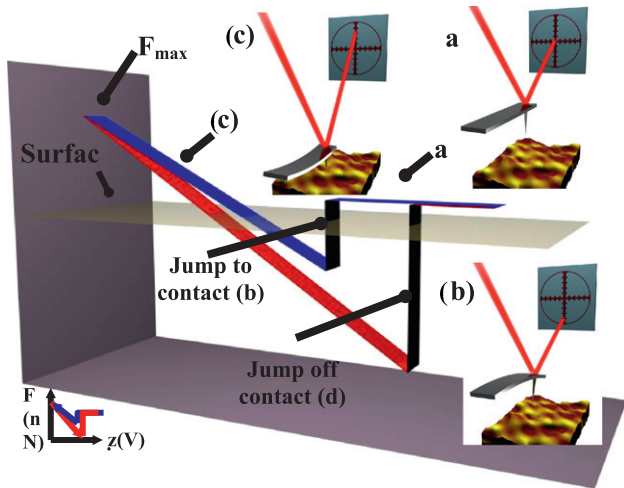


Fig. 1. Schematic of the AFM picoindentation procedure showing the different parts of a typical F - z curve, both in the loading and unloading process: (a) when the tip is far from the surface, there is no interaction between the AFM probe and the sample; (b) as the tip approaches the surface, it bends downwards due to attractive van der Waals forces, the water meniscus or electrostatic forces (*jump to contact*); (c) due to the hard contact between the sample and the surface, the tip bends upwards and the sample is elastically compressed; and (d) during the unloading process, adhesion forces arise between the tip and the sample (*jump off contact*).

(Fig. 1e). This is related to the adhesion forces between the tip and sample during contact.

The first AFM indentations were performed by Soifer et al. [1] on thin film $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ materials, with E values of approximately 210 GPa. However, recent studies performed on $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ samples textured using the Bridgman technique [2], reported E values of 171–181 GPa, while applying loads of between 30 and 100 mN, in agreement with Johansen [3]. Nowadays, different methods are widely used to measure E values for $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ samples and other ceramic materials (ultrasonic techniques [4], X-ray diffraction [5], and bending [6], among others). Nevertheless, it is still difficult to correctly determine an E value without any extra contribution (i.e., in a composite material with inclusions embedded in the matrix it is not possible to correctly determine E value by means of conventional techniques). However, picoindentation measurements are free from any external contribution, and thus the E values calculated using this technique are more accurate and reliable.

Research has clearly demonstrated that residual nanoindentation imprints performed at low penetration depths are greatly affected by surface defects (i.e., surface roughness and inclusions), and this seems to be the reason why E values are so highly dependent on the specific area affected by the indentation [7]. Finally, special attention is paid to the AFM tip behaviour in order to corroborate that the F - z data correspond to the elastic deformation regime of the sample, and that no plastic deformation is experienced by the probe. In this context, the aim of this experimental work is to obtain a new method for determining E values for ceramic materials in the elastic range, without any external contribution, in a straightforward and reproducible way.

2. Materials and methods

2.1. Preparation of bulk YBCO pieces and single crystal growth

2.1.1. Bridgman and top-seeded melt growth technique

The $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ powder was prepared by the polyvinyl alcoholic method (PVA) [8]. The ratio used here (69% w/w Y-123, 30% w/w Y_2BaCuO_5 , Y-211 and 1% w/w CeO_2) was demonstrated to

maximize critical current density [9]. The calcined powder obtained was further deagglomerated by ball milling in an agate mortar.

2.1.1.1. Bridgman technique. Green bulk pieces were obtained by uniaxially isostatic cold pressure and further textured using the Bridgman method [10,11]. Bulk textured pieces were then oxygenated in a vertical furnace at 450 °C for 240 h [11]. From the oxygenated pieces with a common c -axis tilt of 45°, small pieces 2 mm in height were cut along the (0 0 1) plane [12].

2.1.1.2. Top-seeded melt growth technique. The mixture is compressed in the form of a pellet and a seed with cell parameters similar to those of $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ is placed on the top of this pellet. The sample is heated to just above the peritectic temperature, where the mixture is in a semiliquid state. Afterwards, the system is slowly cooled within the right temperature window. If the rate of temperature decrease is slow enough, the mixture is induced to solidify by contact with the solid material that shares its crystallographic orientation with the seed. Thus, the single solid domain expands through the entire sample, which will finally grow with the crystallographic structure induced by the seed. Bulk textured pieces were then oxygenated in a vertical furnace at 450 °C for 240 h [11].

2.1.2. Crystal growth technique

The $\text{YBa}_{1.75}\text{Sr}_{0.25}\text{Cu}_3\text{O}_{7-x}$ [$\text{YBa}(\text{Sr})\text{CuO}$] and $\text{Y}_{0.98}\text{Ca}_{0.02}\text{Ba}_2\text{Cu}_3\text{O}_{7-x}$ [$\text{Y}(\text{Ca})\text{BaCuO}$] single crystals were grown by the self-flux method [13,14]. Y_2O_3 , CaCO_3 , Ba_2CO_3 , SrCO_3 and CuO powders were thoroughly mixed in the proportions 1:3.5:0.5:10 of Y, Ba, Sr and Cu respectively for $\text{YBa}(\text{Sr})\text{CuO}$ samples, and in proportions 0.95:0.05:4:10 of Y, Ca, Ba, Cu for $\text{Y}(\text{Ca})\text{BaCuO}$ samples. Each mixture was piled up on the higher side of a tilted (15°) ZrO_2 tray inside a furnace and slowly heated in air from room temperature to 1020 °C over 4 h. The temperature was maintained at 1020 °C for 1 h and then uniformly cooled to 980 °C over 48 h. Then, the furnace was slowly cooled below 700 °C and finally down to room temperature. In a second round, the $\text{YBa}(\text{Sr})\text{CuO}$ and $\text{Y}(\text{Ca})\text{BaCuO}$ single crystals were treated in pure flowing oxygen at 450 °C for 10 days and then slowly cooled to room temperature.

2.2. Measurement of Young's modulus

2.2.1. AFM topographic measurements

Topographic images were registered in tapping mode in air conditions (40% RH, 20–23 °C) with a 3D-MFP AFM (Asylum Research, Santa Barbara, CA). SiO_2 cantilevers with a nominal spring constant of 40 N/m (ACT-W, Applied Nanostructures, Santa Clara, CA) were used. Force curves were recorded before and after scanning the sample to ensure that the captured images were acquired at minimum vertical force (F) to avoid damaging the sample.

2.2.2. Force microscopy procedure for the calculation of mechanical properties of the samples

Force spectroscopy measurements were performed using the 3D-MFP instrument and the Applied Nanosensors probes cited above. Prior to the experiments, the AFM probes were rinsed with acetone (analysis grade, Merck, WhiteHouse Station, NJ), ethanol (anhydrous RSE for electronic use, Carlo Erba, Rodano, Italy) and Milli-Q water (Millipore, Billerica, MA). Afterwards, the probes were cleaned in a PSD Ultra-Violet/Ozone decontamination unit (Novascan Technologies, Ames, IA). Samples were cleaned using the same procedure except for the water rinsing, as it is known to degrade them. AFM probe radii were measured using the SPIP reconstruction software (Image Metrology, Hørsholm, Denmark) and a SiO_2 test grating with nanometrically sharp ridges (NiOProbe, Aurora Nanodevices, Nanaimo, Canada). This grating is useful to obtain accurate

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