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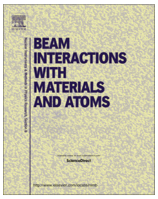

Nanomechanical investigation of ion implanted single crystals – Challenges, possibilities and pitfall traps related to nanoindentation

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ABSTRACT

Nanoindentation technique have developed considerably over last thirty years. Nowadays, commercially available systems offer very precise measurement in nano- and microscale, environmental noise cancelling (or at least noise suppressing), in situ high temperature indentation in controlled atmosphere and vacuum conditions and different additional options, among them dedicated indentation is one of the most popular. Due to its high precision, and ability to measure mechanical properties from very small depths (tens of nm), this technique become quite popular in the nuclear society.

It is known that ion implantation (to some extent) can simulate the influence of neutron flux. However, depth of the material damage is very limited resulting in creation of thin layer of modified material over unmodified bulk. Therefore, only very precise technique, offering possibility to control depth of the measurement can be used to study functional properties of the material. For this reason, nanoindentation technique seems to be a perfect tool to investigate mechanical properties of ion implanted specimens. However, conducting correct nanomechanical experiment and extracting valuable mechanical parameters is not an easy task. In this paper a discussion about the nanoindentation tests performed on ion irradiated YSZ single crystal is presented. The goal of this paper is to discuss possible traps when studying mechanical properties of such materials and thin coatings.

1. Introduction

Nanoindentation method is currently used in many laboratories for evaluation of the mechanical characteristics of the materials [1–3]. One of the biggest advantage of the method is sample requirement: clean, flat and well-polished surface. These requirements give this method an advantage over other conventional methods, such as tension or compression tests. Samples for these experiments require a lot of machining and big quantities of material. These conditions are often difficult to meet, especially when one want to test the conceptual materials produced in small scale and/or limited amount. This is particularly the case for nuclear industry, which very often cannot provide (at least at the early stage of research) large quantities of materials. In addition to that, studied materials must be submitted and tested in the environment similar to true operating conditions of nuclear reactor. This criterion is also very hard to meet because neutrons activate the sample making their handling very difficult. Specially designed Hot Cell Laboratory, dedicated to handle radioactive samples is necessary to conduct these tests. Such tests are very costly, therefore access to such infrastructure is very limited.

Number of studies have shown that ion irradiation can be successfully used to simulate the effects caused by neutron irradiation [4–6]. However, the thickness of the ion irradiated layers usually does not exceed few hundreds of nanometers [7]. Therefore, one could rise a question how to perform valuable analysis of such thin layer on a material which is produced in a limited amount?

Recent works indicate that nanoindentation technique meet all these criteria. For this reason this method is currently one of the most popular methods for mechanical properties evaluation of thin films [8–11]. It is therefore beyond doubt that this technique can be very interested for nuclear engineers working in the domain of ion beam irradiation [12,13]. Since nanoindentation is a perfect tool to study mechanical properties of thin layers, and ion irradiation modify few hundreds of nanometers of the material (at least in the low energy range), the combination of both techniques seems to be a great idea to perform selective material study and investigate hardening effects caused by energetic particles. However, despite relatively easy test procedure, several rules must be complied during the measurement.

For this reason, the aim of this work is to present two most common methodologies of nanoindentation measurement (standard indentation and Load Partial Unload) and to discuss the role of different parameters such us: penetration depth, sample

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roughness, DAF and error statistics. Finally, some examples of mechanical data extracted from ion modified sample will be presented.

2. Experimental

The samples used in this study were commercial Across International, L.L.C. single crystal of YSZ Ytrria Stabilized Zirconia with size 15 × 15 × 0.5 mm. Studied sample was irradiated at room temperature with 160 keV Ar^+ ions up to fluence of 1×10^{16} ions/cm^{-2}. In order to avoid any annealing of the defects due to the target heating, the ion current during irradiation was set as ~0.3 W/cm^{-2} (i.e. flux of the ions was ~1×10^{13} cm^{-2} s^{-1}). The mean projected range of Ar-ions were calculated by using SRIM software [14], and it is ~220 nm.

Nanomechanical investigation were performed by using NanoTest Vantage® Micro Materials Ltd. indentation system. The experiments were performed at room temperature with Berkovich shaped indenter. The indents were made by using three different techniques: (i) line type experiment conducted from 1 to 10 mN of maximum load (i.e.: indents were placed in the line with the distance of 25 μm from each other), (ii) Load Partial Unload (LPU) test mode (i.e.: the sample was loaded in 10 increments up to the maximum load on the same spot. After each load increment, the sample was partially unloaded to 30% of its maximum load and then load increased followed. The procedure was repeated until the maximum load was reached) and finally to study mechanical properties of ion irradiated specimen (iii) by using 3 mN load, which corresponds to approx. 80 nm of maximal indentation depth (~30% of the modified layer). All measurements were performed with 10 s loading time, 1 s dwell period and 5 s unloading time. In order to minimize measurement error related to the sample roughness, each experiment has been repeated at least 10 times and average value is presented. It should be pointed out that during indentation of very thin layer, one must determine the exact shape of the indenter at its top surface. It is known that this part of the tip place a major role during low load nanoindentation and can be potentially source of an error [13]. For this reason, before the experiment, equipment was calibrated and DAF (Diamond Area Function) of the indenter was determined for each applied load. This function was used at every stage of the research. The indents were made by using 1 to 10 mN maximum loads in line type measurement (i.e.: indents were placed in the line with the distance of 50 μm from each other) on Fused Silica sample defined by the following mechanical parameters H – 8.80 GPa, Y – 72 GPa and ν – 0.17 GPa. These parameters were used in the analysis procedure (explained in the further part).

It is known that in order to minimize the effect of the bulk material, the hardness and elastic modulus values should be calculated based on the average data obtained at depths approximately 1/10 of the layer thickness [15]. However, one must remember that this rule is very hard to follow, especially in the case of ion irradiated materials. At the same time several studies have proved that the plastic zone developed under the indenter tip can be up to five times of the indentation depth [16–17]. This effect is less drastic in harder materials with higher yield strength (YS). As reported by Mir et al. [18], high indentation loading results in development of the plastic zone which is 2–3 times thicker than the plastic penetration depth. Due to such big discrepancies among different authors, the only solution seems to be conducting multiple nanomechanical investigation at low load range and average the result. Only by implementing this methodology, one can be sure that calculated mechanical data represent the response of the modified layer (no influence of the bulk material is expected). In order to eliminate creep of the sample, the maximum load should be held for a short time period, for example 1 s. The load/unload curves recorded in nanoindentation experiments were fitted using well known Oliver-Pharr method [19]. One should remember that, the nanoindentation method produces the reduced Young modulus (Er) values, and herein presented Young modulus of the material (E) is obtained by implementing Eq. (1):

$$\frac{1}{E_r} = \frac{1-\nu^2}{E} + \frac{1-\nu_i^2}{E_i} \qquad (1)$$

where E_i and v_i are the Young modulus and the Poisson's ratio of the indenter, respectively 1140 GPa and 0.07. In this study Poisson's ratio of the sample was set as 0.25.

3. Results and discussion

Fig. 1 shows a representative plot of ion concentration in the material developed during low energy ion implantation. It is clearly seen that the highest damage of the material occurs below the surface of the irradiated specimen (black curve). In order to study mechanical properties of modified layer one must perform such nanoindentation test, to collect information only form the modified layer (ideally without response of un-irradiated bulk material – see Fig. 1 dotted black zone). It is known that plastic zone (red curve) developed under indenter tip may by up to 5× thicker than the total penetration depth of the indenter [16–17]. However, according to Mir et al. [18], valuable mechanical parameters may be extracted from the thin layer if the total penetration depth is 3× smaller than the layer thickness. In the presented study, the thickness of modified layer in YSZ is ~220 nm. At the same time indentation depth measured under 3 mN load is ~80 nm. For this reason, one may state that reported results represent response of only ion modified layer (or at least they are burden with the influence of the substrate with minimal extent). In order to tackle problem of nanoindentation depth, which in the case of ion implanted materials is the biggest challenge, control depth indentation procedure should be performed.

It is known that ion irradiation results in radiation damage build-up through creation of the radiation defects and residual stresses in the ion implanted layer [20]. Therefore, one may say that ion irradiation cause formation of a highly metastable atomic

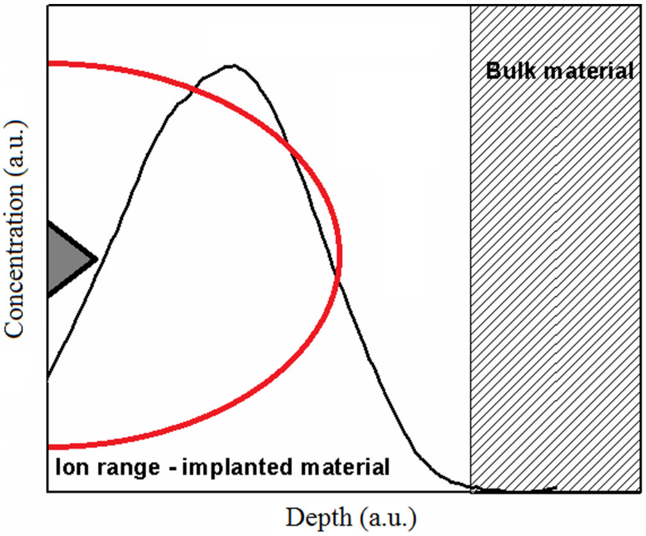

Fig. 1. Representative plot showing the range of low energy ions in YSZ single crystal calculated by SRIM. Black triangle symbolizes indenter and red circle curve depict plastic zone developed under the indenter during measurement (volume from which mechanical characteristics is collected). Dotted region symbolizes unmodified bulk material. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

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