

**1 of 1**

Conf 930928-2

LA-UF- 93 - 2820

Title:

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Submitted to:

Journal of Nuclear Materials

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ISSUES RELATED TO  
MECHANICAL PROPERTIES OF NEUTRON-IRRADIATED CERAMICS

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ABSTRACT

Ceramics will be used for a number of applications in fusion devices, where their dielectric properties, high strength, refractoriness, or low activation characteristics are required. In all cases mechanical properties must be adequate for the use intended, and the material must tolerate irradiation damage without undue degradation. In this paper the damage response of four candidate ceramics is reviewed, major issues are identified, and recommendations made for future studies.

## 1. Introduction

Ceramics are specified for a number of applications in the intense neutron flux of advanced fusion devices such as the International Thermonuclear Experimental Reactor (ITER). Included are electrical insulators for lightly-shielded magnets, toroidal current breaks, rf heating systems, neutral beam injectors, diagnostic devices, and insulating coatings for suppression of MHD forces in liquid metal-cooled blankets. In addition, ceramic optical components are required for some diagnostic systems. Further, ceramics may be specified for first wall protection or for structural applications where low activation characteristics and good high-temperature performance are required.

For all of these applications adequate resistance to radiation-induced mechanical failure during the design lifetime is a necessity. Such failure can result from dimensional changes or a reduction of strength resulting from displacement of lattice ions and their subsequent aggregation. Thus the issues addressed here fall into the broad categories of swelling, reduction of strength, and the microstructural alterations that cause such degradation.

The diversity of applications for ceramics in fusion reactors implies that there is a wide choice among candidate materials, and that is indeed the case. In order to keep this discussion of materials

behavior within bounds, we focus on four candidate materials,  $\text{Al}_2\text{O}_3$ ,  $\text{MgAl}_2\text{O}_4$ ,  $\text{SiC}$  and  $\text{SiO}_2$ , assessing the response of each to neutron irradiation. Taken together, the behavior of this set of materials is illustrative as to the types of damage response that can be expected for most ceramics in fusion applications.

## 2. Neutron Damage in Four Candidate Ceramics

### 2.1 $\text{Al}_2\text{O}_3$

$\text{Al}_2\text{O}_3$  is probably the most commonly-specified ceramic insulator where good structural and electrical performance are required. With respect to fusion requirements, this material in either its polycrystalline form (alumina) or single-crystal form (sapphire) is a candidate for most applications. The crystal structure of this ceramic is hexagonal. The greatest shortcoming of  $\text{Al}_2\text{O}_3$  with respect to fusion applications is its relatively high sensitivity to neutron damage.

Irradiation of this ceramic to a fluence of  $2 \times 10^{26}$  n/m<sup>2</sup> near room temperature results in near-isotropic swelling on the order of 2 vol.% [1]; such growth is believed to result primarily from accumulation of point defects. Irradiation at higher temperatures leads to greater swelling, with most of the growth being in the c-axis direction (Fig.1). This behavior has been shown to result from

preferential aggregation of displaced atoms in interstitial loops having Burgers vectors that contribute primarily to growth perpendicular to basal planes [2].

This preference for c-axis dilation leads to macroscopic distortion of single crystals, and can present problems in developing mounting systems for sapphire windows for diagnostic applications. This behavior also presents problems for the polycrystalline form, in which each grain grows anisotropically. As a result alumina irradiated at elevated temperatures can suffer a buildup of internal strains, in some cases leading to grain boundary microcracking or separation. [1,2].

Strength of ceramics is represented by the relationship

$$\sigma_f = \text{const.} \frac{\sqrt{E\gamma}}{\sqrt{c}} = \text{const.} \frac{K_{Ic}}{\sqrt{c}},$$

where  $\sigma_f$  = fracture strength, E = Young's modulus,  $\gamma$  = fracture energy, c = critical flaw size, and  $K_{Ic}$  = fracture toughness. The grain boundary microcracking or separation observed in alumina represents an increase in flaw size, and can therefore lead to weakening unless other flaws (e.g., those from fabrication) dominate. This equation does not take into account internal stresses, which for alumina will result both from anisotropic thermal contraction upon cooling from the sintering temperature and from anisotropic swelling; such stresses will have a weakening effect on this ceramic.

Fracture toughness is affected by interaction of propagating cracks with the damage microstructure. As mentioned earlier, irradiation near room temperature results primarily in the accumulation of point defects. Irradiation to a fluence of  $2 \times 10^{26}$  n/m<sup>2</sup> at 430 K leads to formation of a dense array of small loops [2]. Exposure to a similar fluence at 925 and 1100 K induces a microstructure consisting of dense dislocation networks (formed by the interaction of interstitial loops) and aligned voids [2].

Fracture toughness of sapphire has been found to be doubled by irradiation at 1100 K, an effect attributed to interaction of the crack tip with voids of diameter 9 nm [3]. Results for sapphire and two other ceramics (AlN and SiC) after irradiation to fluences up to  $4 \times 10^{26}$  n/m<sup>2</sup> at moderate to high temperatures also show increases, although of a lesser magnitude [4]

Room-temperature strength measurements on single-crystal Al<sub>2</sub>O<sub>3</sub> exhibit considerable variation. This material showed a moderate decrease of strength after irradiation to  $2 \times 10^{25}$  n/m<sup>2</sup> at 373-388 K [4] and a similar decrease after a fluence of  $2 \times 10^{26}$  n/m<sup>2</sup> at 673-873 K [5] (Fig. 2); however, no significant change was observed after irradiation at 680 and 815 K [6]. It is unfortunate that no results are available at higher temperatures where void formation has been reported.

Polycrystalline  $\text{Al}_2\text{O}_3$  irradiated to  $2 \times 10^{25}$  n/m<sup>2</sup> at 373-388 K showed a moderate decrease of strength [4]. That result is consistent with strength losses reported for other grades of alumina irradiated to ten times higher fluence at 673-873 K [5] (Fig. 2) and is similar to results for another alumina after  $2 \times 10^{26}$  n/m<sup>2</sup> at 660 K [7]. In contrast, Colin [8] found an 80% strength loss after a fluence of  $5 \times 10^{25}$  n/m<sup>2</sup> at 823 K.

The role of grain size in determining extent of strength loss in alumina has not yet been resolved: Wilks et al. [9] presented calculations indicating that a fine grain size should lessen internal strains and postpone microcracking to higher fluences, and Keilholtz et al [1] found that their finest-grained material was most resistant to grain boundary cracking. On the other hand, Dienst [5] saw no major difference in damage response between materials of grain size 0.9 and 8 microns.

## 2.2. $\text{MgAl}_2\text{O}_4$

Magnesium aluminate spinel is an electrically-insulating ceramic with reasonably good mechanical properties, and is usable in most applications for which  $\text{Al}_2\text{O}_3$  is a candidate material. The greatest virtue of  $\text{MgAl}_2\text{O}_4$  is its good performance under neutron irradiation.

Spinel is for the most part a low-swelling ceramic. Dimensional changes are near-zero for both single-crystal and polycrystalline material after irradiation to fluences on the order of  $1 \times 10^{26}$  n/m<sup>2</sup> [2], with the exception of 0.8 vol.% swelling at 430 K [10] and 0.2 to 1.6 vol.% at 925-1100 K [2]; both of the exceptions are for the polycrystalline form. Recent measurements on single-crystal and polycrystalline material irradiated at temperatures of 658, 678, and 1023 K to fluences as high as  $24 \times 10^{26}$  n/m<sup>2</sup> have shown that dimensional stability extends even to that high damage level [11].

It has been proposed that the characteristic low swelling of spinel is attributable to predominant recombination of point defects rather than their aggregation; the open crystal structure with its large number of structural vacancies, along with the capability to intermix the site occupancy of Mg and Al ions, may also play a role in the dimensional stability of this ceramic [2]. Further, the fact that spinel has a cubic crystal structure means that anisotropic swelling is not an issue in this material.

The most common response of spinel in either single-crystal or polycrystalline form to neutron irradiation is to exhibit strengthening [4,6,7,10] (Fig. 2). However, it must be pointed out that Colin [8] found a large decrease of strength in his spinel material. It has been proposed [6] that the strengthening behavior results from impedance of crack propagation by the damage microstructure, specifically by

the intense strain fields around interstitial dislocation loops. Another explanation for the observed strengthening has been suggested, namely the blunting of atomically-sharp tips of pre-existing flaws by displacement damage [12].

The damage microstructure of polycrystalline spinel differs from that in single-crystal material in ways that may explain the lesser strengthening observed in the polycrystalline form [6]. After irradiation at 430 K [10] and 660 K [7], areas near the grain boundaries are found to be denuded of the characteristic interstitial loops that are found within the grains. Such a damage microstructure may induce misfit stresses near grain boundaries that work against the inherent strengthening effect that apparently operates in the bulk material.

### 2.3 SiC

Silicon carbide is a high-strength ceramic. In most forms this material is a semiconductor rather than an insulator, and therefore its principal use for fusion reactors is as a structural material. Possible applications include use as first wall protection, as reflectors in diagnostic systems, and as structural members where good high-temperature mechanical properties and/or low nuclear activation are desired. SiC is available in either the alpha (hexagonal crystal structure) or beta (cubic) form.

The swelling behavior of pyrolytic beta SiC from room temperature to 1313 K has been characterized by Price [13,14]. Swelling of this cubic material exhibited saturation between  $1 \times 10^{24}$  and  $4 \times 10^{25}$  n/m<sup>2</sup>, and decreased from 3 vol. % at the lowest temperature to near zero at the highest. Lack of a difference between macroscopic linear expansion and lattice parameter expansion implied that most of the swelling was due to the creation of point defects or small clusters. Work by others [6] subsequently showed that swelling in beta-SiC irradiated to  $2 \times 10^{26}$  n/m<sup>2</sup> at 680 K was of the same magnitude as that reported by Price [13], thus demonstrating that saturation persists at least up to that fluence. At irradiation temperatures of 1523 and 1773 K Price observed void formation and significant swelling [14]. However, growth did not achieve the levels measured at room temperature.

Strength of irradiated silicon carbide is strongly dependent on the form of the material tested. Price found that pyrolytic beta SiC exhibited no significant change in strength after irradiation to 2 to  $4 \times 10^{25}$  n/m<sup>2</sup> at temperatures ranging from 733 to 1313 K. [13], and the same was true for that material after a fluence of  $2 \times 10^{26}$  n/m<sup>2</sup> at 1013 K [15]. However, strength of sintered alpha SiC was reduced 34% by the same irradiation conditions, and reaction-bonded SiC was weakened by 58%. It was proposed that weakening of the sintered material resulted from transmutation of the boron sintering aid to helium through the  $^{10}\text{B}(n,\alpha)$  reaction, which resulted in an

increase of critical flaw size [15]. The larger strength reduction for the reaction-bonded SiC was attributed to dissimilar swelling between the SiC and the 8 to 10% free silicon that was present in that material.

Strength measurements for SiC show some disagreement: Dienst [5] has reported strength losses on the order of 60% for both isostatically hot-pressed (HIP) and chemically vapor-deposited SiC (alpha and beta phase, respectively) after irradiation to  $2 \times 10^{26}$  n/m<sup>2</sup> at 773 to 873 K. The behavior of the HIP material (Fig. 2) could be due to anisotropic swelling, but the observed differences in high-dose behavior of beta SiC [5,15] remain unexplained.

## 2.4 SiO<sub>2</sub>

Silicon dioxide is commonly used in both the crystalline form (usually quartz, with a trigonal or hexagonal structure) and the glassy form (vitreous or fused silica). Both exhibit modest strength but have excellent electrical and optical properties. SiO<sub>2</sub> may be used in a variety of diagnostic applications, including windows, substrates for reflectors, and optical fibers; in addition, silicate-based ceramics may, because of their fabricability, be specified for the large neutral beam injector ion source insulators. This material is unique among the four discussed here in that both forms can undergo atomic displacements from ionizing radiation (e.g., gamma

rays), although Hobbs and Pascucci [16] have shown that this mode of damage is relatively inefficient in SiO<sub>2</sub>.

Both crystalline and glassy SiO<sub>2</sub> undergo large changes in density at low neutron fluences (Fig. 3), with quartz swelling and fused silica densifying to a common value [17]. In the case of quartz, atomic displacements rearrange Si and O atoms into the disordered or metamict state. The atoms of fused silica are already in a disordered state before irradiation damage, but that condition is different from the metamict state; the effect of neutron damage is to transform the original amorphous structure to the metamict form that is unique to irradiated material.

Little information is available on mechanical properties of either crystalline or glassy SiO<sub>2</sub>, before or after irradiation. Strength of these materials is on the order of 100 to 200 MPa, with fracture toughness of approximately 1 MPa m<sup>1/2</sup>. The authors are not aware of any strength measurements on either form of SiO<sub>2</sub> under irradiation conditions that approximate those to be found in a fusion reactor. However, neutron damage in a machinable glass-ceramic made up of 50 vol.% silicate mica crystals in a silicate glass matrix has been investigated after irradiation to fluences of 1x10<sup>22</sup> and 1x10<sup>23</sup> 14 MeV n/m<sup>2</sup> at room temperature [18,19]. Density and fracture toughness were essentially unaltered after exposure to the lower fluence; however, as fluence increased to the higher dose,

swelling first reached a value of 1.6 vol.% and then lessened to a value half that level. It was concluded that this unusual behavior resulted from swelling of the mica phase and contraction of the glass phase [19].

The greatest concern with respect to the use of quartz, and to a lesser extent fused silica, is the large dimensional changes at low radiation levels. Such behavior in a free-standing material does not in itself threaten diagnostic systems except for possible warpage of reflectors. However, any component whose design involves mechanical constraint, as in the case of optical fibers and sealed windows, will suffer high stresses and the possibility of fracture. Analysis of expected dilation and resulting stresses in quartz and fused silica windows mounted in window assemblies suggests an upper fluence limit of about  $5 \times 10^{21}$  n/m<sup>2</sup>, unless sliding seal assemblies can be developed [20].

### 3. Major issues

The above review of dimensional, microstructural, and mechanical property changes in four candidate materials illustrates the variety and complexity of damage responses likely to be encountered. We now take a broader view of the subject, identifying what we believe to be the major issues in this area.

Two problems result from the current necessity of employing fission neutrons to achieve high damage levels in materials to be used in fusion reactors: the probability that primary damage events from fission and fusion neutrons differ significantly, and the fact that greater concentrations of transmutation products will be generated by high-energy fusion neutrons. Of these two problems, the latter is probably the more significant. Rovner and Hopkins [21] have calculated the levels of gaseous and metallic transmutation products that will be generated in four ceramics under first wall conditions (Table 1); it may be shown that, given a realistic first wall loading and lifetime, impurity levels on the order of 1 at.% can be expected. The effect of these transmutation products on physical properties can only be surmised, but given the effect on SiC of helium generated by transmutation of boron sintering aid [15,22], there is cause for concern.

It has been observed that values of Weibull modulus for  $Al_2O_3$  [5] and SiC [15,22] are significantly reduced by neutron damage. Such a broadening of strength distribution means that even a material with an acceptably high average strength under laboratory conditions may when fabricated into components exhibit an unacceptably high failure rate. Proof testing of unirradiated components to eliminate those most likely to fail will not necessarily be of significant help [15]. Thus a high Weibull modulus can be as

important a property as high strength, and must be given more attention than has been the case to date.

Some non-cubic ceramics suffer from anisotropic swelling of individual grains, with consequent generation of internal stresses and possible grain boundary separation. As discussed earlier, swelling anisotropy and grain boundary separation have been observed for alumina [1,2,9], and could play a role in irradiation-induced degradation of other non-cubic ceramics such as AlN, Si<sub>3</sub>N<sub>4</sub>, and alpha-phase SiC.. There has been no work quantifying the reduction of strength that is expected to accompany anisotropic swelling, although the severe microcracking observed by Keilholtz et al. [1] indicates qualitatively that this can be a significant problem. On the other hand, the observation by Dienst [5] that fine-grained Al<sub>2</sub>O<sub>3</sub> suffers a strength reduction similar to that for coarse-grained material and that single-crystal sapphire shows a decrease of strength similar to that for polycrystalline materials indicates that other factors can mask the consequences of anisotropic swelling.

A little-considered source of premature strength loss in technical ceramics is the presence of a damage-sensitive grain boundary phase. Such a situation has been encountered in sialon [23], where a silicate grain boundary phase was found to exhibit significantly more damage (in the form of pore-like aggregates) than did the bulk material. When damage-sensitive grain boundary

phases are identified, it may be possible to alleviate the problem by making appropriate adjustments to the composition of the bulk material.

Some applications for ceramics in fusion devices involve bonded or coated systems, e.g., protective tiles for the first wall, reflectors for diagnostic applications, and an insulating lining for piping in liquid metal-cooled blankets. A major potential problem area for these applications is differential swelling, which can lead to high interfacial stresses, warpage, and delamination or bulk fracture. An example is the response of a layered system consisting of a thick coating of chemically vapor-deposited beta SiC on graphite: after irradiation to  $2 \times 10^{26}$  n/m<sup>2</sup> at 680 K [6] the material was visibly bowed, and, and the interface had almost completely separated. It was found that the SiC had swelled 1.5 vol.% while the graphite substrate had densified 7 vol.%. The lesson here is that monolithic ceramics should be used whenever possible, and where a layered system is unavoidable the design should if possible employ a thin coating of deformable material as one of the components. Composite materials are also at risk from differential swelling. Where such materials are required, the best choice would be a two-phase ceramic in which the phases are as similar as possible; an example is bulk SiC reinforced with SiC fibers or whiskers, where toughening can be realized through interfacial debonding under applied stress.

Swelling under thermal and flux gradients may generate internal stresses that can reduce the load-bearing capability of irradiated ceramics. This is not expected to be a major problem for test samples or small components, but could prove to be a significant issue for large components such as neutral beam injector ion source insulators. Given an adequate data base, it should be possible to calculate the magnitude of macroscopic internal stresses and determine whether these stresses present a problem.

#### 4. Recommended future studies

As a general statement, we can say that work to date on mechanical properties of neutron-irradiated ceramics has identified major areas of concern, developed a useful if incomplete data base, and in some areas has established an understanding of basic damage effects. However, more effort is needed in these areas if ceramics with adequate performance and lifetime are to be available on a timely basis for construction of fusion reactors such as ITER.

At the fundamental level, an understanding is needed of primary damage events in ceramics. To achieve this understanding, modeling calculations and experiments should be carried out to follow the evolution of damage from cascade events involving time scales of a few atomic vibrations, (i.e. on the order of a picosecond) to the relaxed state that evolves over perhaps the next microsecond.

Knowledge from such studies can guide the selection of critical experiments to define the evolution of a quasi-steady-state defect content that is characteristic of the material, test temperature, irradiation fluence, and dose rate.

Given an adequate understanding of damage effects, it should be possible to predict how generic classes of ceramics differ in their response to radiation damage and use that as a guide to selecting the most promising candidate materials. Examples of where some understanding already exists are the differences in damage response between ionically-bonded and covalently-bonded ceramics, and the propensity of certain crystal structures to support annihilation of vacancies and interstitials rather than their aggregation. Greater understanding in these areas could reduce the amount of time and effort spent in evaluating what might prove to be unpromising materials.

It is well known that surface finish plays an important role in determining the critical flaw that propagates to cause fracture of a ceramic. For this reason, special care should be devoted to attaining a uniform and well-characterized surface condition both for test samples and for components themselves.

Studies of mechanical properties should be conducted in the appropriate test environment. Examples are an SF<sub>6</sub> atmosphere for

testing of rf windows and the appropriate liquid metal for studies of MHD-suppressing insulators. Since designs of fusion devices can be expected to continually evolve, it is incumbent on designers and materials researchers to communicate frequently in order to assure that tests are being conducted in the proper environment.

For non-cubic ceramics, swelling and orientation of applied stresses should be assessed with proper attention to crystallographic direction. The same attention to orientation is important for the understanding of the role of defect aggregates, in this case for both cubic and non-cubic materials.

Transmutation gases are an important part of the damage condition for ceramics irradiated with high-energy neutrons. More experiments are needed that combine high-fluence damage with concurrent production of hydrogen and helium, using techniques such as isotopic tailoring to attain appropriate gas generation rates [24].

Little effort is currently being directed to development of improved ceramics for fusion applications. The present state of understanding of neutron damage effects is now adequate to support increased activity in this area, and so communication between researchers and manufacturers should be established now in order to

assure timely development of qualified ceramic materials for ITER and beyond.

### Acknowledgements

The authors would like to thank the U.S. Department of Energy and the European Fusion Technology Program for their support of this work.

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**Table 1**  
**Major transmutation products induced in ceramics by first wall neutrons (appm/y at a first wall loading of 1 MW/m<sup>2</sup>) [21]**

	SiC	Si <sub>3</sub> N <sub>4</sub>	Al <sub>2</sub> O <sub>3</sub>	BeO
<b>Hydrogen</b>	440	667	456	103
<b>Carbon</b>	–	328	624	569
<b>Helium</b>	1595	758	787	2920
<b>Magnesium</b>	458	455	434	–

## Figure Captions

Figure 1. Dilation along a- and c-axes in neutron-irradiated single-crystal  $\text{Al}_2\text{O}_3$  as a function of irradiation temperature [2].

Figure 2. Strength change of various  $\text{Al}_2\text{O}_3$  grades including single-crystal samples (SC), HIP-SiC, and  $\text{MgAl}_2\text{O}_4$  after neutron irradiation at 673-873 K [5]; and the same materials after irradiation at about 380 K (data band around  $2 \times 10^{25}$  n/m<sup>2</sup>) [4].

Figure 3. Density change in alpha-quartz and vitreous  $\text{SiO}_2$  neutron-irradiated near room temperature [17].

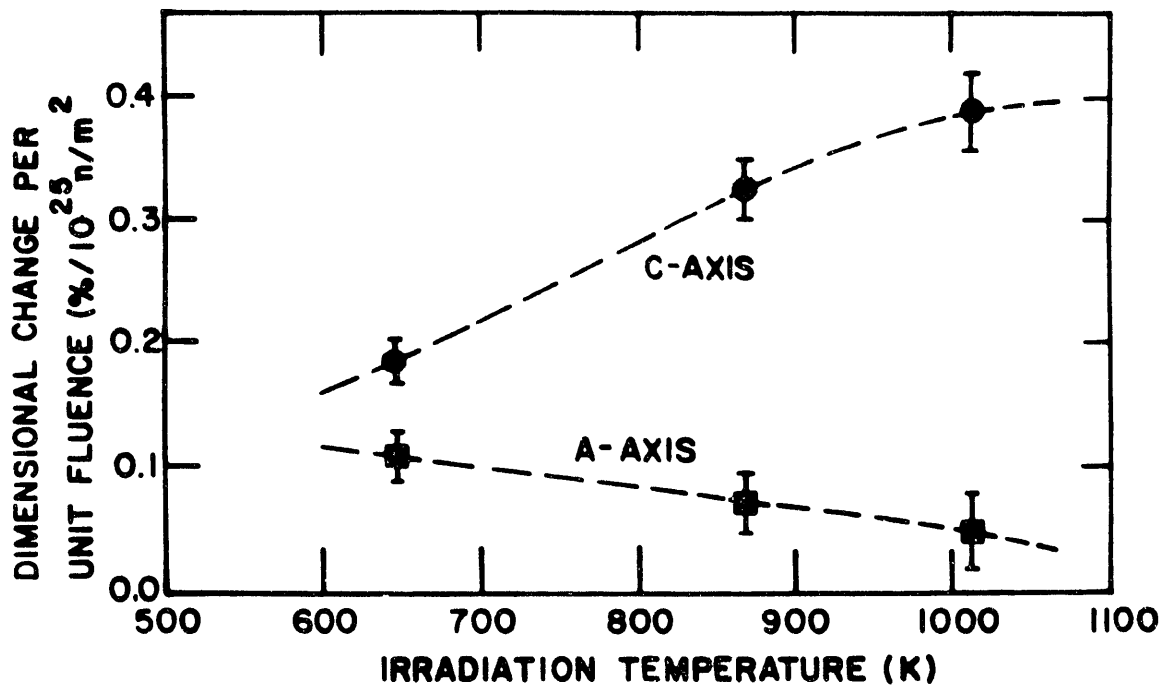


Fig. 1

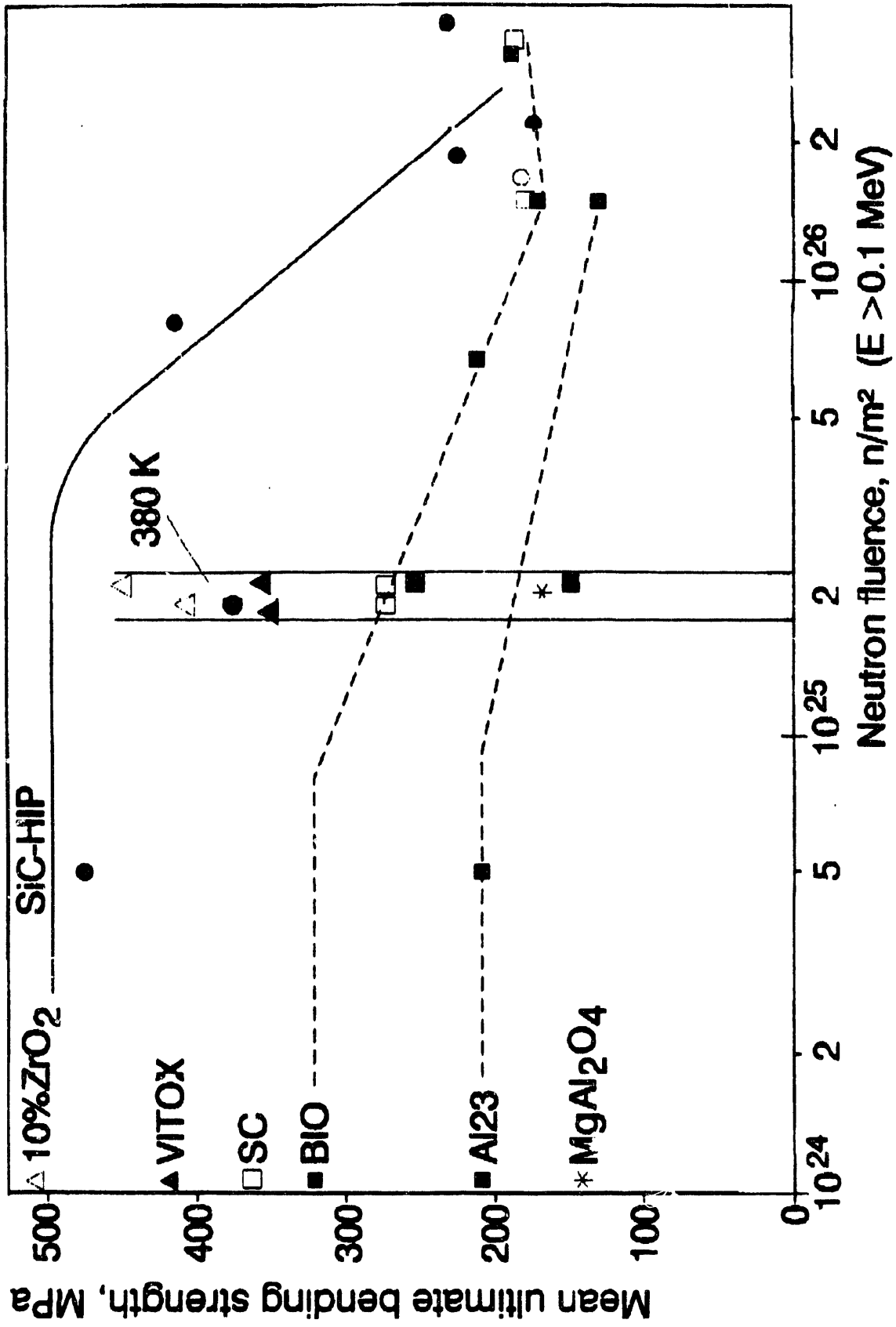


Fig. 2

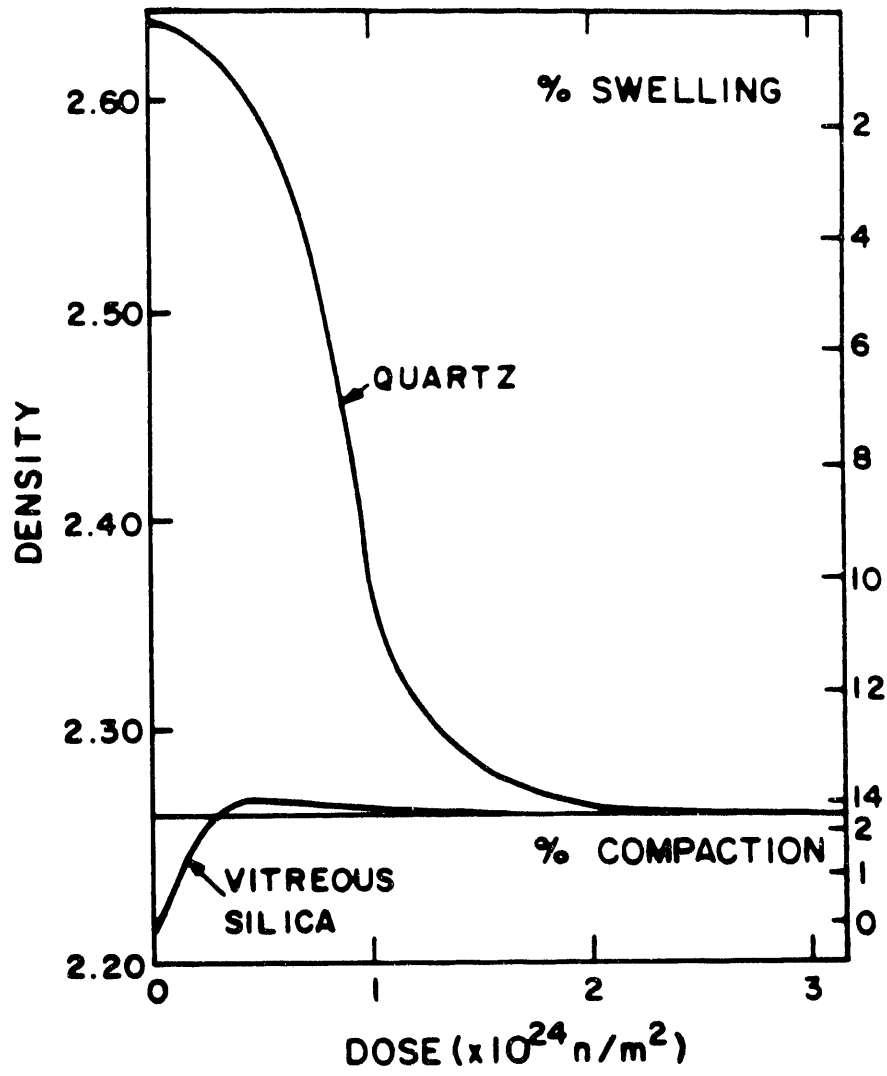


Fig. 3

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