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IRRADIATION EFFECTS IN BeO AND COMPARISON OF BeO AND MgO
AS POTENTIAL REFLECTOR MATERIALS

Title: UNCLASSIFIED

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December 21, 1967

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TO: Distribution

FROM: G. Sidhu

SUBJECT: Irradiation Effects in BeO and Comparison of BeO and MgO as Potential Reflector Materials

Introduction

The nuclear, physical, chemical and mechanical properties of BeO make it an attractive material for use as a reflector or a moderator, particularly at higher temperatures. The material has high scattering and low absorption cross-sections for neutrons. It also possesses satisfactory strength, good thermal conductivity and thermal-shock resistance and low chemical reactivity, etc. For these reasons BeO has been chosen as a reflector material in various reactor designs.

Fairly high neutron fluxes may be expected in the proximity of the reflector of a space power reactor. It is therefore necessary to estimate the possible changes in the essential properties of the reflector material in the radiation environment and establish its integrity even after large neutron doses. Some estimates of degradation of BeO characteristics can be made from the experimental information currently available in the literature⁽¹⁾. But for an uncharacterized specimen such information may serve merely as a qualitative guide unless all the variables are taken into account. The experimental results depend not only on neutron flux, radiation dose and temperature, as in any irradiation study, but also on such factors as the starting material, method of fabrication, impurities, grain size, porosity and density. Some of the effects of radiation on BeO, such as increase of volume, migration and agglomeration of transmutation products (chiefly helium), defect clusters, microcracking, changes in elastic constants and mechanical strength, are

discussed here without experimental details. The numerical values presented here, however, should be considered only as guides rather than quantitatively reliable. Some steps to minimize the problem of radiation damage and possible use of MgO as a reflector are also suggested.

Dimensional Changes During Irradiation

Irradiation by neutrons causes an increase in volume of BeO. Such growth is a function of several variables, notably temperature of irradiation, radiation dose, neutron flux, material density, grain size, impurities and method of fabrication. The change of volume may be attributed to some or all of the following processes.

Defect Clusters in BeO Lattice

Interstitials, vacancies and transmutation products are continuously generated in BeO due to irradiation by neutrons, although some annealing does take place. The defects diffuse through the BeO lattice to form small ($< 10^{\circ}\text{\AA}$) or large clusters. Some of the large clusters may grow into dislocation loops.

The anisotropic expansion behavior of BeO is attributed to its crystal structure which is like that of wurtzite (Zn S). The structure can be described as a hexagonal closed packed sublattice of oxygen ions with a similar and interpenetrating sublattice of beryllium ions. The lattice parameter in the basal plane (regular hexagon) of the crystal is denoted by "a" and that in a direction normal to the plane is referred to as "c". It is the way the small and large defect clusters tend to arrange themselves in the lattice that gives the observed growth behavior of BeO.

Small defect clusters, which anneal easily in the range below 800°C , constitute a major portion of the total concentration at low radiation doses ($\sim 10^{19}$ nvt) and low temperatures⁽²⁾ ($\sim 100^{\circ}\text{C}$). At higher doses ($\sim 10^{20}$ nvt) they constitute a relatively small proportion of total defect concentration and their concentration saturates at about 5×10^{20} nvt. These small clusters (from one to several defects each) are believed to be responsible for the "a" parameter change.

At higher doses the large clusters dominate; they do not anneal as readily as small clusters do. These large clusters arrange themselves parallel to the basal planes of the hexagonal lattice and are believed to be responsible for the large change in the "c" parameter. Their concentration, and hence the growth along the "c" axis, does not saturate as readily as the concentration of small clusters does.

Anisotropic Lattice Expansion and Microcracking

The preferential arrangement of large defect clusters in a BeO crystal, leads to the observed anisotropic lattice expansion, i.e., unequal growth along the "c" and the "a" axes. If the grains in a BeO specimen acquire preferred orientation during manufacture the whole specimen grows anisotropically. At $\sim 100^{\circ}\text{C}$ the change in "c" axis is about 8 times that in the "a" axis and the latter saturates at about 0.15%. At low temperatures ($\sim 100^{\circ}\text{C}$) the volume change for a certain specimen of BeO has been measured to be 0.2% to 0.3% per 10^{20} nvt dose⁽³⁾. At a dose of about 2×10^{20} nvt the expansion rate for a 5μ grain size specimen has been observed to increase by a factor of about 3 due to microcracking⁽³⁾. The term microcracking is used to describe the separation at the grain boundaries of a polycrystalline material, resulting in the creation of voids in the

specimen; less frequently a few intragranular microcracks have also been observed. Microcracking is caused by the stresses generated by the anisotropic growth of randomly oriented grains in polycrystalline BeO. This microcracking is the principal contributor to the expansion of BeO at low temperatures and leads to some drastic changes in its physical and mechanical properties. The dose at which microcracking begins and the degree of microcracking for a certain dose level depend on the microstructure of the specimen and on the irradiation temperature. Thus small grain size specimens are more resistant to microcracking and at higher temperatures annealing inhibits microcracking. At $\sim 100^{\circ}\text{C}$, BeO specimens of 20μ grain size have been reported to have become friable at $\sim 8 \times 10^{20}$ nvt ($\sim 6\%$ expansion) and disintegrated to powder at $\sim 10^{21}$ nvt. The same dose at 1000°C causes no serious mechanical damage.

Helium Bubbles

At higher temperatures the growth due to lattice expansion and microcracking is very much reduced, but the bubbles of helium gas may make a significant contribution to the volume increase. Nuclear reactions such as $\text{Be}^9(n, \text{He}^4) \text{He}^6 \xrightarrow{\beta} \text{Li}^6$ and $\text{Be}^9(n, 2n) \text{Be}^8 \rightarrow 2\text{He}^4$ produce comparatively large numbers of helium atoms which, being inert, tend to diffuse and agglomerate to form bubbles at higher temperatures. The coalescence and resultant increase in volume of these bubbles must exert pressure on the surrounding structure and cause an increase in volume of the material. Generally this effect is more pronounced at higher temperatures ($\sim 1000^{\circ}\text{C}$), when bubble formation occurs more rapidly. According to Collins⁽³⁾ the gas bubbles in a BeO-0.5 wt% MgO specimen of 15μ grain size and density of

2.9 irradiated to $\sim 10^{21}$ nvt at 950°C were responsible for a volume increase of about 0.4%, which is about half the total volume increase. But later estimates in other irradiation tests at $\sim 1000^{\circ}\text{C}$ suggest⁽⁴⁾ that most of the expansion at this temperature and high doses may be attributed to helium gas bubbles. For specimens of typically 1/4" radius the estimates of gas escaped during irradiation range from 20% to 50% for doses of the order of 10^{21} nvt.

Experimental Information on Volume Increase

The volume increase at low temperatures is mainly due to microcracking. At higher temperatures, due to some annealing of defects and consequently reduced lattice expansion, microcracking is very much reduced and does not appear until higher neutron doses. But at a temperature of $\sim 1000^{\circ}\text{C}$ the expansion due to helium may become dominant. A plot of volume change vs. neutron dose for a BeO specimen of 5μ grain size and density 2.9 g/cm^3 irradiated at 1000°C indicates a volume increase of about 0.6% per 10^{21} nvt dose⁽⁴⁾ (see Fig. 1). The linearity of growth vs. neutron dose will probably not continue at doses above 10^{22} nvt when some saturation of defect concentration may occur. For the lower density (2.6 g/cm^3) material the maximum total expansion has been reported⁽⁴⁾ to be about 3% as shown in Fig. 2. For comparison a plot of irradiation growth at low temperature is reproduced in Fig. 3 from earlier work⁽³⁾. The results of experiments at Oak Ridge⁽⁵⁾, shown in Fig. 4, further illustrate the effects of temperature, material density and grain size, but little dependence of expansion on neutron flux has been reported. Some of the results of irradiation studies in BeO at G. E. are reproduced

Fig. 1. Volume expansion as a function of exposure in BeO specimens with a grain size of $\sim 5 \mu$ and a density of 2.9 g/cm^3 irradiated at 1000°C (Ref. 4).

Fig. 2. Volume expansion as a function of dosage in BeO specimens with a grain size of $\sim 5 \mu$ and a density of 2.6 g/cm^3 irradiated at 1000°C (Ref. 4).

Fig. 3. Volume expansion in different grain sizes and densities of AOX composition of BeO at 100°C (Ref. 3).

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Fig. 4. Volume Increase of 1/2-in. BeO Specimens vs Fast-Neutron Dose (Ref. 5).

from the G. E. Report GEMP-61 (1966). (In the table AOX and UOX refer to different grades of BeO made by Brush Beryllium Co. Small quantities of MgO or ZrO_2 or some other material may sometimes be added to improve the sintering characteristics and bulk properties of BeO.)

Changes in Mechanical Properties

Microcracking, helium bubbles and other radiation effects, which cause a change in density of BeO, likewise affect its mechanical properties. The effect is roughly proportional to growth in volume. Therefore volume increase is usually taken as a fair criterion for fast neutron damage, except for cases where the volume increase may be due to chemical reactions such as oxidation of impurities.

At low doses and high temperatures, i.e. for small expansion before microcracking can occur, the rupture strength is essentially unchanged and in fact may increase. Young's and shear moduli also decrease very little. But when microcracking sets in, the rupture strength begins to decrease rapidly and at higher doses may lead to fracturing and powdering of the material. In a 20μ grain-size specimen of density 2.9 g/cm^3 irradiated to varying neutron doses of up to 1.5×10^{21} at temperatures between 100 to 1000°C , the decrease in rupture strength began⁽³⁾ at a volume expansion of about 0.5%. This threshold was nearly independent of temperature although it took higher doses at higher temperatures to produce the same expansion. But the strength was down by a factor of 10 when the expansion reached about 2% and deteriorated further at higher volume changes until the material became brittle, friable and turned to powder. The smaller the grain size of the material the more resistant it is to expansion and the higher the strength it retains under given irradiation

Some of the Tabulated Data from Ref. 4.

SUMMARY OF RADIATION EFFECTS IN BaO SPECIMENS IRRADIATED AT 1000°C TO 6 TO 9 x 10²¹ nvt (E_n ≥ 1 Mev)

Sample Description		Irradiation Conditions ^a				Expansion					Increase in Percent Open Porosity	Young's Modulus		Modulus-of-Rupture at Room Temperature	
		1st Test	2nd Test	Total		1st Test	2nd Test	Total				10 ¹² dyne/cm ²	Percent of Unirradiated	10 ³ kg/cm ² ^b	Percent of Unirradiated Value
Composition	Grain Size, microns	Dosage, 10 ²¹ nvt (E _n ≥ 1 Mev)	Flux, 10 ¹⁴ av (E _n ≥ 1 Mev)	Dosage, 10 ²¹ nvt (E _n ≥ 1 Mev)	Dosage, 10 ²¹ nvt (E _n ≥ 1 Mev)	Volume, %	Length, %	Dia., %	Volume, %	Volume, %					
UOX-MgO	5	2.88	1.1	2.1	5.6	0.6	1.1	1.0	3.1	3.7	1.6	3.14	89	2.56, 2.67	120, 125
	5	2.88	1.2	2.6	7.0	0.7	1.4	1.3	4.1	4.8	4.3	3.08	87	2.56, 2.58	121
	5	2.88	0.8	2.0	7.7	0.5	1.7	1.4	4.5	5.0	5.3	3.12	86	2.63, 2.28	123, 107
	6	2.91	-	2.6	7.1	-	1.6	1.3	4.1	4.1	4.6	3.23	89	2.72, 2.68	128, 126
	6	2.74	1.1	2.6	7.1	0.7	1.2	1.1	3.4	4.0	3.9	2.88	86	2.53, 2.59	124, 127
	4	2.74	1.0	2.0	7.7	0.8	0.9	1.0	2.8	3.7	7.1	2.93	90	2.26, 2.20	111, 109
	4	2.74	1.0	2.0	7.8	1.0	0.7	0.7	2.1	2.1	7.8	2.94	88	1.87, 1.92	92, 94
	8	2.56	1.1	2.1	5.6	0.7	0.7	0.6	1.9	2.9	12.8	2.56	88	1.16, 1.63	55, 76
	6	2.56	1.3	2.4	6.6	7.0	1.0	0.6	0.5	1.6	12.7	2.55	88	1.90, 1.61	88, 75
	6	2.56	1.1	2.7	7.4	8.5	0.8	0.6	0.7	2.3	13.3	2.50	86	1.35, 1.45	63, 67
	6	2.56	1.2	2.9	7.8	9.0	0.9	0.6	0.6	2.1	13.4	2.29	79	-	-
	6	2.56	1.0	2.9	7.9	8.9	0.8	0.7	2.2	3.0	13.4	2.52	87	1.31, 1.73	60, 80
	12	2.90	1.2	2.3	6.3	7.5	0.9	1.2	0.5	2.3	3.3	3.33	90	2.05, 2.16	94, 93
	12	2.90	1.1	2.8	7.5	8.6	1.0	1.0	0.7	2.0	2.9	3.13	84	1.46, 1.38	67, 63
	18	2.90	1.1	2.3	6.3	7.4	1.0	2.0	0.8	2.6	2.4	2.44	83	0.58, 0.36	26, 16
	8	2.90	-	1.7	4.5	4.5	-	0.9	1.0	2.8	1.3	3.25	89 ^c	2.28, 1.74	108, 82
	8	2.90	-	2.6	7.0	7.0	-	2.0	2.4	6.8	6.0	-	-	0.85, 0.82	40, 39
	8	2.90	-	2.6	7.1	7.1	-	2.1	2.3	6.7	2.2	2.46	71 ^c	1.14, 1.27	54, 50
AOX	8	2.90	-	2.9	7.8	7.8	-	1.8	1.6	4.9	2.0	3.03	88 ^c	1.86, 1.62	68, 66
	2.4	2.78	1.1	1.7	4.6	5.6	1.1	0.3	0.3	0.9	4.6	-	-	1.93, 2.15	109, 120
	2.4	2.78	0.9	2.1	6.6	6.5	1.2	0.3	0.4	1.1	4.3	2.36	73	0.71, 1.39	40, 78
	2.4	2.78	1.1	2.4	6.6	7.7	1.0	0.3	0.3	0.9	4.1	-	-	-	-
	2.4	2.78	1.0	2.8	7.5	8.5	0.9	0.7	0.7	2.1	5.7	3.94	93	1.06	60
	2.4	2.78	1.1	2.9	7.9	8.9	0.7	0.7	0.8	2.3	7.1	2.74	86	1.58, 1.82	89, 102
	6	2.80	-	2.7	7.4	7.4	-	1.7	2.3	6.3	7.6	-	-	0.60, 0.80	38, 32
	6	2.80	-	2.9	7.8	7.8	-	1.9	1.2	3.3	4.0	3.08	93	2.11, 1.69	100, 80
	6	2.80	-	2.9	7.9	7.9	-	1.6	2.0	5.6	6.6	-	-	0.78, 0.75	37, 37
	5	2.57	0.9	2.7	7.4	8.3	0.9	0.7	0.8	2.3	12.3	2.48	87	-	-
	5	2.57	1.0	2.7	7.5	8.5	0.9	1.0	1.1	3.3	13.2	1.92	67	0.90, 1.07	44, 52
	5	2.57	1.0	2.9	7.8	8.8	0.9	0.9	1.1	3.0	13.7	1.95	54	0.83, 0.79	41, 39
	17	2.29	-	2.3	6.3	6.3	-	1.3	1.7	4.8	4.3	-	-	0.78, 0.60	33, 25
	17	2.89	1.0	2.4	6.6	7.6	1.2	0.8	0.9	2.6	2.9	2.40	86	1.00, 1.13	42, 47
UOX-3ZrO ₂	2.9	2.91	-	1.7	4.5	4.5	-	0.7	0.6	1.9	4.7	3.08	90 ^c	3.10, 2.74	125, 111
	2.9	2.91	-	2.6	7.0	7.0	-	1.8	1.3	4.4	2.3	3.13	88 ^c	2.00, 2.85	81, 116
	2.9	2.91	-	2.9	7.7	7.7	-	1.0	0.8	2.6	6.5	3.01	92 ^c	2.43, 2.25	98, 91
	2.9	2.91	-	2.9	7.8	7.8	-	0.9	0.7	2.3	5.2	3.06	90 ^c	2.64, 1.95	107, 79

^aSpecimens were irradiated, examined, then irradiated further in a second test.

^bStrength values listed are 0.8 of the values measured with 3-point loading over a 2.22 cm span on specimens of 0.6 cm diameter; this value approximates that obtained with 4-point loading over a 7.62 cm span.

^cThese values are based on comparison to the average value for specimens of the same composition and sintering batch rather than pre- and post-irradiation measurements of the same specimen.

conditions. Although low density specimens are believed to retain strength better than high density ones different reports^(3,6,7) do not agree regarding the effect of density on resistance to mechanical damage. The tabulated data reproduced here from Ref. 4 shows results for different specimens and experimental conditions.

Thermal Conductivity

Thermal conductivity of unirradiated BeO at 1000°C is an order of magnitude lower than that at room temperature⁽⁸⁾. But on irradiation to a few times 10^{20} nvt at room temperature the thermal conductivity is reduced by an order of magnitude whereas it is virtually unchanged after irradiation by a similar dose at 1000°C, presumably due to annealing of defects. According to experimental observations the large planar defect clusters can be nearly completely removed by annealing⁽⁸⁾ at 1100°C. This implies that helium bubble formation at higher temperatures does not seriously reduce the thermal conductivity of BeO, which is nearly equal to its unirradiated value. Experimental data are tabulated in detail in Ref. 4 and 5.

Implications for SPR Reflector Design

For a space power reactor the neutron flux at the reflector may be expected to be of the order of 10^{14} nv. If the temperature of the BeO reflector is low, say below 600°C, the increase in volume and decrease in strength could cause fracture and brittleness within a few weeks. Since the temperature of the reflector is radiation limited and will probably not be below 1000°C, a well selected and fabricated reflector material can survive without serious damage up to a dose of 4×10^{21} nvt. Accord-

ing to G. E. work⁽⁴⁾ most fine-grained BeO specimens irradiated at $\sim 1000^{\circ}\text{C}$. to $\sim 8 \times 10^{21}$ nvt retained 60 to 80% of their rupture strength. A few additional comments relevant to our reactor program can be made on the basis of the above discussion.

1. Even for total neutron doses of only $\sim 4 \times 10^{21}$ nvt during reactor operation at $\sim 1000^{\circ}\text{C}$, a reflector of small grain size BeO ($\leq 2\mu$) fabricated with the best available techniques will be necessary. Although reports on damage resistance vs density of BeO are conflicting, low density material, which suffers smaller volume change, should be preferable. For a certain dose one would expect somewhat greater deterioration at higher fluxes but experimenters report no noticeable flux effects⁽⁵⁾. There is a decrease in thermal conductivity of BeO by an order of magnitude at $\sim 1000^{\circ}\text{C}$ compared to that at room temperature. This should not be a serious handicap because of the compensatory effect of greater annealing and lower damage at higher temperatures and practically no further decrease of conductivity due to irradiation at high temperatures. Finally, the thickness of a reflector is expected to be a few times greater than that of the BeO specimens usually tested. But in irradiation tests, fine-grain specimens with larger dimensions do not show any greater damage than those with smaller specimens. So, even though some stresses in larger volumes of BeO may be expected due to higher temperatures and lower expansion in central portions, the problem is not likely to be serious.

2. For a reflector where a dose of only about 10^{21} nvt is expected and the operating temperature is $\sim 1000^{\circ}\text{C}$, BeO should present no serious problem. If the material is fabricated with proper microstructure, negligible

decrease in strength and less than 0.5% expansion in volume should result for this dose and any flux below $\sim 4 \times 10^{14}$ nv may be considered safe.

3. If the neutron dose is expected to exceed $\sim 10^{22}$ nvt at $\sim 1000^\circ\text{C}$, some refractory material cladding for BeO should be considered, to keep the reflector from disintegrating at these neutron doses. Since the gas released as a result of nuclear reactions in the reflector will be mostly helium, any pockets of the gas between BeO and the cladding material would not reduce thermal conduction to the surface.

MgO as Potential Reflector Material

The idea of an MgO reflector, as an alternative to BeO may be worth studying. The neutron scattering cross-section of Mg, for the SNAP-50 flux spectrum, is higher than that of Be. So the macroscopic scattering cross section in MgO should be comparable to that in BeO. But the capture cross section of Mg is lower than that of Be by a factor of 6 or 7. Further, the neutrons scattered by MgO will be degraded somewhat less than those scattered by BeO. This may have the desirable effect of reducing the sharp spike in the power profile (due to the absorption of the reflected neutrons) usually present at the periphery of the fuel volume. It is also possible that in this intermediate energy region the capture/fission ratio may be lower for the less degraded neutrons from Mg. In SPN-90 the reflector worth of BeO in SPR-3 is shown to be greater than that of MgO. For the SPR-6 reactor, B. Koponen has shown in SPN-145 that up to a reflector thickness of 8 cm BeO is better. However, for thicknesses larger than 8 cm, MgO has been shown to be superior. These problems need further investigation and it is quite likely that the neutronic properties of an MgO reflector will compare favorably with those of BeO.

No helium is generated by transmutations in Mg at the neutron energies of interest. This would make MgO potentially more useful at higher temperatures where inert gas bubbles may cause materials problems. The only other transmutation product in Mg, hydrogen, should not have any really deleterious effects.

The comparison of mechanical and thermal properties* of MgO and BeO is difficult because of the sensitivity of their properties to grain size, density, impurities and the methods of fabrication. Thermal properties of MgO may be only slightly worse than those of BeO. Specific heat of MgO at room temperature is about the same as that of BeO but at 1000°C becomes about 60% of the latter although both increase with temperature.

At ~1000°C the thermal expansion coefficient of MgO is 20 to 30% higher than that of BeO. Thermal conductivities of both materials decrease at higher temperatures and at ~1000°C the ratio K_{MgO}/K_{Be} is about one-half. BeO must retain a better thermal shock resistance at these temperatures because of its higher conductivity. But the higher temperature in the interior of the reflector due to the poorer conductivity of MgO should not be a serious handicap and there is always the benefit of reduced radiation damage at higher temperatures in the absence of inert gas bubbles in MgO.

The mechanical properties of MgO also come fairly close to those of BeO. The bend strength of MgO at room temperature is about the same as that of BeO but at 1000°C may reduce to half of that for a comparable specimen of the latter. The compressive strengths of the two at ~1000°C

* This information is taken from "Engineering Properties of Ceramics", Technical Report AFML-TR-66-52 by J. F. Lynch, et al., Battelle Memorial Institute.

are about the same although at low temperatures BeO is better. Their tensile strengths at 1000°C are also comparable although MgO may be superior at room temperature. For the two materials Young's, shear, and bulk moduli are comparable, within a factor of 2. Again, the creep rate and hardness, although highly dependent on microstructure and methods of fabrication, are usually within a factor of two for comparable specimens. Thus although more detailed investigation of the physical properties of MgO may be necessary, it seems like a good reflector material at first sight.

The main reason for the choice of MgO may be its irradiation properties. Unlike BeO, this material does not suffer anisotropic growth (MgO crystal structure is believed to be cubic which may partly account for the difference). As a result the grain boundary separation due to microcracking does not occur. At $\sim 100^{\circ}\text{C}$ the growth in MgO is much smaller^(9,10) and tends to saturate at $\sim 1\%$ for higher doses⁽¹⁰⁾. At $\sim 1000^{\circ}\text{C}$ the fractional growth in MgO is smaller than that in BeO by an order of magnitude⁽⁹⁾ after a dose of about 4×10^{21} , although according to Ref. (10) the difference is about two orders of magnitude. Irradiation tests by Sambell and Bradley⁽¹¹⁾ indicate that the stress at which plastic flow occurs in MgO is increased from 10 Kg/mm^2 to $\sim 130 \text{ Kg/mm}^2$ after irradiation by $\sim 10^{20}$ nvt.

Increased damage and fracture during irradiation in MgO is reported in Ref. (9) at higher temperatures compared to that at intermediate temperatures, in spite of annealing. This could not be attributed primarily to thermal effects because such fracturing did not occur in out-of-pile thermal cycling or long-term heating. Thermal stresses due to gamma heating have been suspected, and of course the cause could also be

some defective specimen since no serious damage in MgO is reported in Ref.

9.

The current information on properties of MgO suggests that its use as a reflector material is worth consideration.

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