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MATERIALS RESEARCH AND DEVELOPMENT FOR FUSION ENERGY APPLICATIONS

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Some of the critical issues associated with materials selection for proposed magnetic fusion reactors are reviewed, with a brief overview of refractory alloys (vanadium, tantalum, molybdenum, tungsten) and primary emphasis on ceramic materials. SiC/SiC composites are under consideration for the first wall and blanket structure, and dielectric insulators will be used for the heating, control and diagnostic measurement of the fusion plasma. Key issues for SiC/SiC composites include radiation-induced degradation in the strength and thermal conductivity. Recent work has focused on the development of radiation-resistant fibers and fiber/matrix interfaces (porous SiC, SiC multilayers) which would also produce improved SiC/SiC performance for applications such as heat engines and aerospace components. The key physical parameters for dielectrics include electrical conductivity, dielectric loss tangent and thermal conductivity. Ionizing radiation can increase the electrical conductivity of insulators by many orders of magnitude, and surface leakage currents can compromise the performance of some fusion energy components. Irradiation can cause a pronounced degradation in the loss tangent and thermal conductivity. Fundamental physical parameter measurements on ceramics which are of interest for both fusion and non-fusion applications are discussed.

1. INTRODUCTION

Fusion energy is considered to be an attractive future large-scale energy source due to the large quantities of available fuel (e.g., hydrogen isotopes) and passive safety features (fusion is a driven reaction, i.e., the fuel reaction can be immediately stopped in case of an accident). Magnetic fusion reactors will require a diverse array of materials in order to achieve the desired operating conditions. These include high heat flux materials, high strength structural materials, tritium breeding materials, optically transparent materials, low-loss dielectrics and electrical insulators, and superconducting magnet materials. All of these materials must exhibit adequate resistance to neutron radiation damage effects during the plant lifetime. The projected operating environment near the plasma is particularly severe, with heat fluxes of ~1 to 20 MW/m^2 and neutron fluxes of ~5 to 30x10^{18} n/m^2-s.

Many of the key issues associated with the selection of materials for magnetic fusion energy have been discussed in the literature.[1-5] The current status of ferritic/martensitic steels for the reactor structure is reviewed in a companion paper in these proceedings.[6] The present paper will briefly discuss the status of research and development on

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vanadium and high temperature refractory alloys for fusion structural applications, and then will review some of the recent progress and critical issues associated with structural (SiC/SiC) and dielectric ceramics.

2. REFRACTORY ALLOYS

The high heat fluxes adjacent to the plasma can induce large temperature gradients and corresponding thermal stresses in the first wall and blanket structural materials. Copper alloys such as CuCrZr, CuNiBe, CuCrNb and oxide dispersion strengthened copper have a high thermal stress figure of merit at temperatures below ~300°C ($M=\sigma_U k_{th}(1-\nu)/\alpha E$ ~30-60, where σ_U is the ultimate tensile strength, k_{th} is the thermal conductivity, ν is Poisson's ratio, α is the coefficient of thermal expansion, and E is Young's modulus). However, the achievable power plant thermal efficiency is relatively low since copper alloys cannot be used above ~400°C due to thermal creep.[7]

Refractory structural alloys (W, Mo, Ta, Nb, V) are attractive for energy production due to the potential for high thermal efficiency associated with operation at high temperatures. In addition, some refractory metals such as W- and Mo-alloys are under consideration for high heat flux plasma-facing components such as the divertor due to their high thermal conductivity and low chemical and physical sputtering. The thermal stress figure of merit for these refractory alloys varies from 6 to 12, which is higher than the corresponding values of M=2 to 5 for austenitic and ferritic/martensitic steels. All of these refractory alloys have problems associated with oxygen pickup and oxidation at temperatures above ~500°C.[8] This requires special precautions with many of the potential coolants in order to avoid oxygen contamination and is a main reason for the current relatively small market for these alloys. The current status of R&D on refractory alloys for non-fusion applications has been reviewed by several authors.[9-11]

Figure 1 compares the ultimate tensile strengths of Cu-Ni-Be and Fe-8-9%Cr martensitic steel with several refractory alloys. The refractory alloys exhibit superior tensile strengths at temperatures above 600°C. The refractory alloys are typically used in a stress-relieved condition in order to take advantage of the higher strength (and improved ductility in the case of Mo and W) compared to recrystallized alloys. However, the possibility of stress- or radiation-enhanced recrystallization does not allow this strength advantage to be considered for conservative structural engineering analyses. Recrystallized strengths were used in Fig. 1 for the refractory alloys.

All of the refractory alloys appear to have good compatibility with potential coolants such as Li and Pb-17Li, although further corrosion studies on these materials are still needed at high temperatures.[12-14] However, Mo and W alloys are difficult to fabricate at ambient temperatures. The ductile to brittle transition temperature (DBTT) of these alloys is typically above room temperature,[8] and neutron irradiation at temperatures up to ~700°C causes a further increase in the DBTT.[15] This "low-temperature" embrittlement is

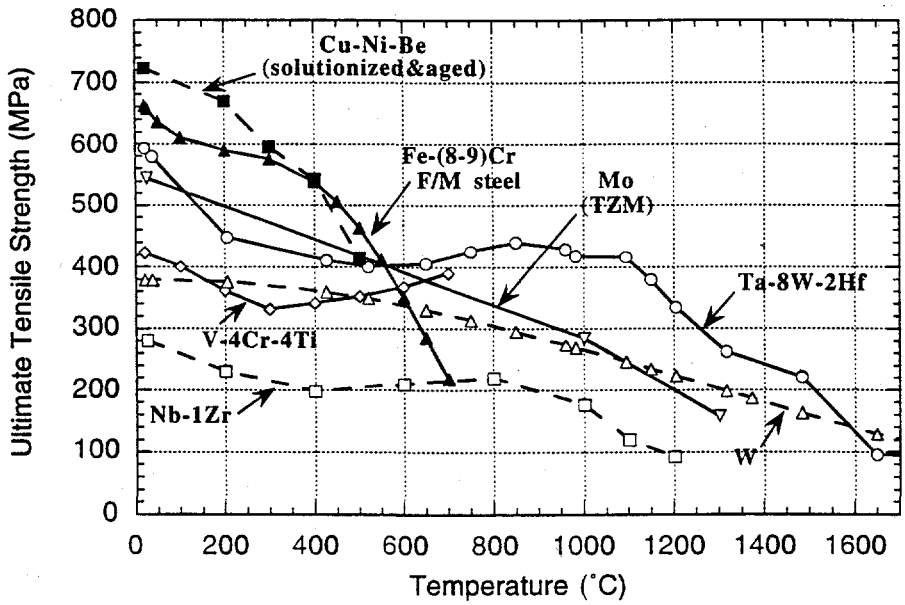

Fig. 1. Comparison of the ultimate tensile strengths of recrystallized refractory alloys[8,16-19] vs. solutionized and aged Cu-2Ni-0.3Be[20] and martensitic Fe-(8-9)Cr steel.[21]

one of the main obstacles for using Mo and W alloys as structural materials in a fusion reactor. Additional concerns for the refractory alloys include high short-term decay heat in irradiated W and Ta alloys and long-term induced radioactivity in Mo and Nb alloys.

Alloys based on the Group Vb elements (V, Nb, Ta) generally have significantly better fabricability compared to Mo and W alloys, with DBTTs below room temperature.[8] There has been considerable interest in vanadium alloys for fusion energy structural applications, due to their attractive combination of moderate strength (up to ~700°C), adequate fabricability, good ductility above room temperature, and low long-term induced radioactivity. The current status of research and development on vanadium alloys for fusion applications (in particular V-4Cr-4Ti) has been recently reviewed.[22] Low temperature radiation embrittlement will likely limit the lower operating temperature of V-4Cr-4Ti alloys to ~400°C. The maximum operating temperature limit for vanadium alloys is expected to be ~700°C (0.45 T_M) due to thermal creep, although the existing data base[19] is very sparse at temperatures above 600°C. The operating temperature windows for other refractory alloys are estimated to be 650-1300°C for Ta alloys, 750-1200°C for Mo alloys, and 800-1400°C for W alloys, based on radiation hardening, thermal creep and chemical compatibility issues.

3. SiC/SiC COMPOSITES

SiC/SiC composites have received increasing attention for fusion reactor structural applications in the past decade, due to their high temperature strength, acceptable fracture toughness (~20-30 MPa-m$^{1/2}$) and low induced radioactivity and afterheat.[4,23-25] The identified key issues include challenges associated with scale-up fabrication of large complex components, the lack of suitable standardized techniques for joining SiC/SiC composite parts (including field repairs), concerns about effects of matrix porosity and microcracking on coolant leak rates at pressure boundaries (particularly for He coolant), the need for standardized engineering design criteria for ceramic composites, and high costs for currently available composites. Chemical compatibility studies indicate that the oxidation rate for SiC/SiC in He-cooled systems should be acceptably low up to 1000°C, if graphite interphases are not used.[24] Limited studies[26,27] suggest that SiC may corrode in liquid Li at temperatures above 500°C, but it is stable in stagnant Pb-17Li at 800°C.

SiC/SiC composites are composed of 3 different components: fiber, matrix, and interphase. Work performed to date on SiC/SiC composites has been hampered by the lack of large quantities of high-quality SiC-based fibers. As summarized in Table 1, the chemical composition and properties of early commercial SiC-based fibers (cg-Nicalon, Hi-Nicalon) are significantly different from bulk SiC. More recent SiC based fibers (Hi-Nicalon-S, Dow Sylramic, MER CVR fiber) have compositions and properties closer to that of bulk SiC, but are currently only available in limited quantities.

Table 1. Comparison of properties of commercial SiC-based fibers and bulk SiC.[28-30]

	cg-Nicalon	Hi-Nicalon	Hi-Nicalon type S	Dow Sylramic	Bulk SiC
Diameter (μm)	14	12-14	12	10	----
Tensile strength (GPa)	2.0-3.0	2.8-3.4	2.6-2.7	2.8-3.4	~0.1
Elastic modulus (GPa)	170-220	270	420	390-400	460
Density (g/cm^3)	2.55	2.74	2.98-3.10	3.0-3.10	3.25
Coefficient of thermal expansion (10^{-6}/K)	3.2	3.5	---	5.4	4.0
Oxygen content (wt.%)	11.7	0.5	0.2	0.8	0.0
C/Si atomic ratio	1.31	1.39	1.05	1.0	1.0

The interphase between the fibers and matrix is of critical importance for high fracture toughness. It must be weak enough to inhibit matrix crack propagation by promoting fiber-matrix debonding and fiber sliding, and sufficiently strong to transfer load between the matrix and fibers in order to take advantage of the high fiber strength. Early work on SiC/SiC composites typically used a ~0.2 μm graphitic interphase. However, this

interphase has poor oxidation resistance at elevated temperatures. Several advanced interphase materials are currently being investigated,[31] including porous SiC and multilayer SiC. Figure 2 compares the flexure strength behavior of SiC/SiC composites fabricated with several types of interphases.[31] Room temperature flexure properties comparable to that obtained with optimized graphite interfaces have been achieved. In addition to improved high temperature oxidation resistance, these advanced interphases exhibit improved radiation resistance compared to a graphite interphase.[31] In particular, the matrix microcracking stress was unchanged after irradiation to ~1 dpa at 300-1000°C. These advanced interphases would also be beneficial for SiC/SiC composites in demanding non-fusion applications such as heat engines and aerospace components.

Fusion researchers have focused on several other fusion-specific issues, including radiation effects on thermal conductivity, mechanical properties, and dimensional stability. The minimum and maximum operating temperature of SiC in a fusion reactor will likely be controlled by radiation effects. Significant volumetric swelling (>3%) has been observed[31] in SiC irradiated at temperatures ≥1000°C. A very large volumetric swelling of ~12% occurs in SiC at neutron irradiation temperatures below ~140°C due to amorphization.[31]

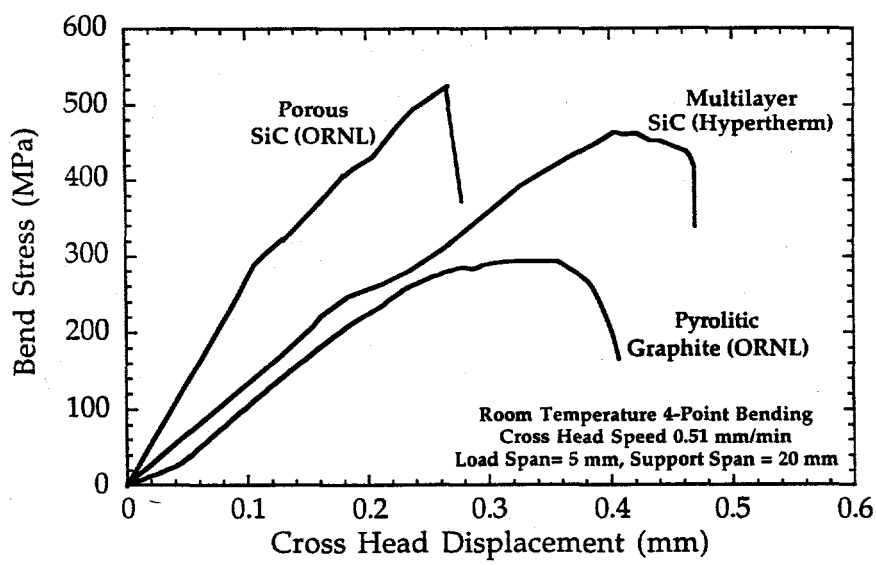

Fig. 2. Comparison of the flexure bar behavior of SiC/SiC (Hi-Nicalon fibers, forced CVI matrix) composites fabricated with three different types of interphase.[31] Note that the 0.4 μm graphite interphase was not optimized for maximum strength.

One important consequence of the large chemical differences between currently available SiC-based fibers and pure SiC (Table 1) is that that the fibers and stoichiometric SiC matrix in the composite will respond differently to irradiation. Figure 3 shows the effect of neutron irradiation on the density of SiC and SiC-based fibers at 500-650°C. A moderate amount of swelling (~1.2%) occurs in pure SiC during irradiation, with an apparent saturation in swelling observed at doses above ~0.1 displacements per atom (dpa). In contrast, both ceramic grade Nicalon and Hi-Nicalon experience significant densification, with the largest densification observed in the lower-grade cg-Nicalon fibers. The data in Fig. 3 and other studies[29,32-34] indicate that amount of densification in cg-Nicalon increases with increasing irradiation temperature between 150 and 810°C. The shrinkage of the fibers during irradiation causes debonding with the matrix, and produces low strength in irradiated composites fabricated using current grades of SiC-based fibers. The typical decrease in strength observed in irradiated composites is ≥20%, depending on the irradiation conditions.[4,31,32] Smaller strength changes would be expected in irradiated composites containing advanced fibers (due to a better match with the SiC matrix behavior), but experimental data are not yet available.

The optimal performance of SiC/SiC composites in a high power density fusion reactor would be critically dependent on the thermal conductivity. The thermal conductivity (k_{th}) of SiC/SiC composites is strongly dependent on the processing conditions, type of fiber, and fiber architecture. High purity monolithic SiC represents the upper limit for k_{th} in SiC/SiC composites, with maximum values of ~320 and 78 W/m-K at 20 and 1000°C, respectively (Fig. 4). Currently available SiC/SiC composites have much

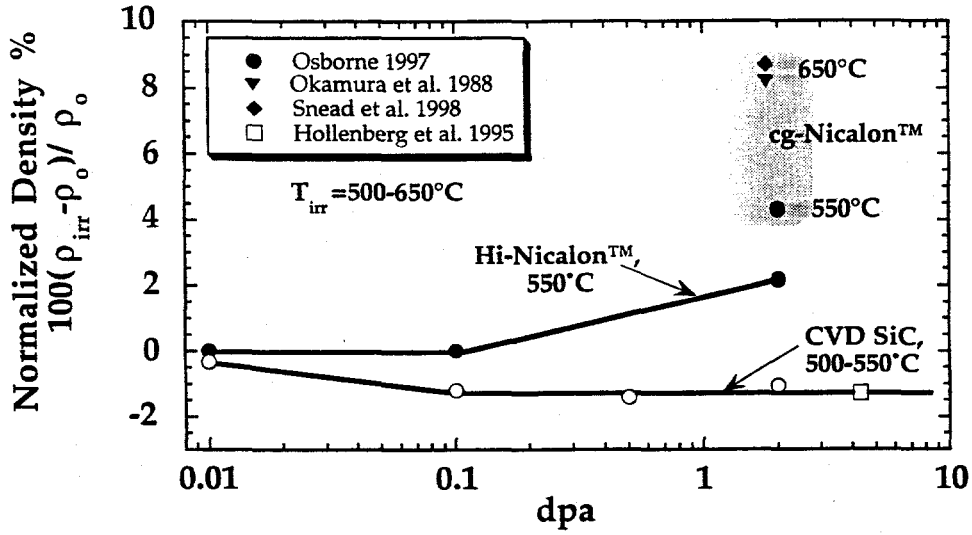

Fig. 3. Effect of neutron irradiation on the density of SiC and SiC-based fibers.[29,31,32,35]

lower thermal conductivity than high-purity monolithic SiC, primarily due to a combination of poor quality fibers (k_{th}~3 to 5 W/m-K in cg- and Hi-Nicalon), matrix microcracking and infiltration porosity (boundary scattering), and imprecise control of the CVI deposition chemistry for the matrix. Commercial SiC/SiC composites have typical cross-ply and in-plane conductivities of ~10 and ~30 W/m-K, respectively. SiC/SiC composites with cross-ply conductivities of ~75 W/m-K at 20°C and 30-35 W/m-K at 1000°C have recently been fabricated using CVR and reaction sintering techniques.[36,37]

Neutron irradiation causes a significant decrease in the thermal conductivity of non-metals, due to increased phonon scattering by the radiation-produced defects. Figure 4 indicates that the irradiation reduces the thermal conductivity of monolithic SiC to ~15 W/m-K at temperatures near 400°C, and ~20 W/m-K at 1000°C. This radiation-induced degradation makes it unlikely that the thermal conductivity of SiC/SiC composites would exceed 15 W/m-K in a fusion reactor, even if further advances in the processing of unirradiated composites are achieved. Further thermal conductivity data on irradiated monolithic SiC and SiC/SiC composites are needed to investigate this issue.

A crystalline to amorphous phase transition occurs in SiC during irradiation at low temperatures (below ~150°C). Amorphization cannot be tolerated in structural applications, due to the large volumetric swelling of ~12% that accompanies this phase transition.[31] Using the assumption that amorphization is due to increases in free energy

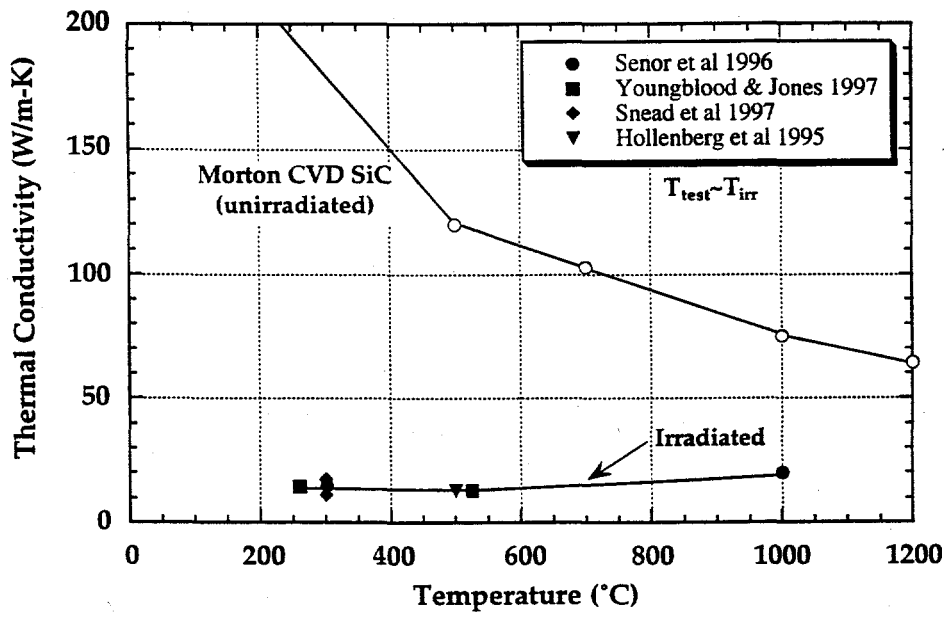

Fig. 4. Summary of thermal conductivity data on unirradiated and neutron-irradiated monolithic SiC.[32,38-40]

associated with point defect accumulation, an estimate of the migration enthalpy for the rate-controlling (slowest) interstitial species of ~0.85 eV has been obtained from an examination of the temperature-dependent amorphization behavior of SiC irradiated with different particles.[41] Estimations of the vacancy and interstitial migration enthalpies in SiC have large variations, ranging from 1 to 3 eV for vacancy migration and ~1 to 4 eV for interstitial migration.[42-45] Further work is needed to obtain more accurate estimations of these fundamental parameters.

4. DIELECTRICS

Ceramic insulators are required in numerous components for the heating, control, and diagnostic measurement of fusion plasmas. The ionizing radiation fields near the first wall of a fusion reactor would be ~10^4 Gy/s, which is sufficient to cause large increases in electrical conductivity and modest increases in the dielectric loss tangent at frequencies $\leq$100 MHz.[46,47] Figure 5 summarizes radiation-induced conductivity (RIC) measurements in Al_2O_3. The RIC is approximately proportional to the ionizing dose rate at temperatures <500°C. Similar RIC data have been obtained in other insulating ceramics, including $MgAl_2O_4$, MgO, Si_3N_4 and AlN.[46-48] The existing data base indicates that the magnitude of the RIC in SiO_2 is about a factor of ten lower than in Al_2O_3.

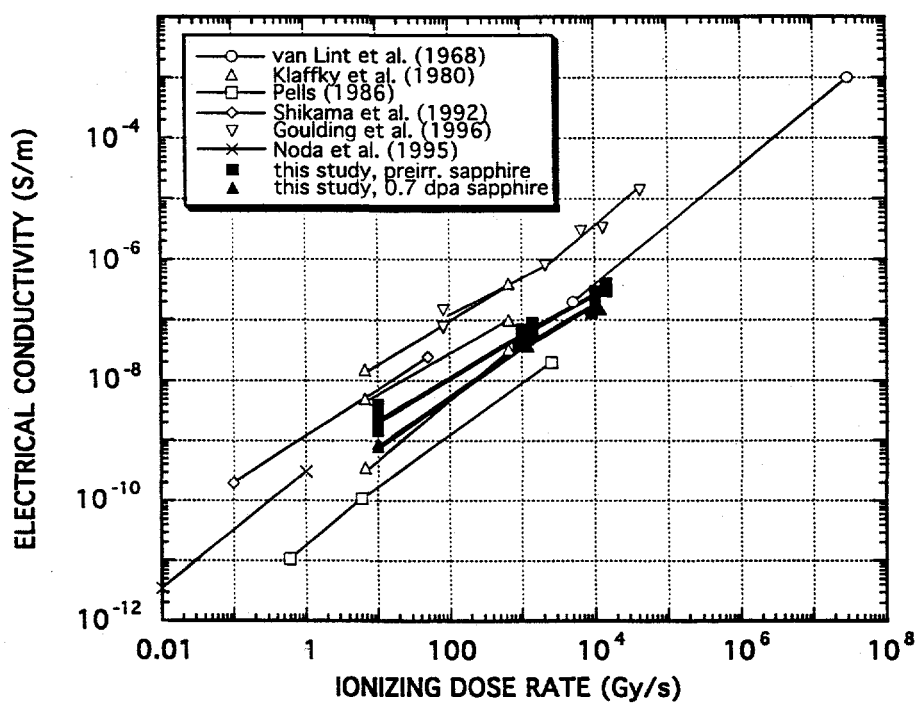

Fig. 5. Electrical conductivity of Al_2O_3 measured during particle irradiation.[49]

Considerable attention has recently been focused on the possibility that, in addition to the well-known transient increases in electrical conductivity (RIC), a permanent increase in the electrical conductivity may occur if the ceramic insulator is irradiated with an applied electric field.[3,46] Most of the evidence for this permanent radiation induced electrical degradation (RIED) has been obtained from electron irradiations.[3,46,50] However, recent ion[51] and neutron[49] irradiation experiments have failed to observe the RIED phenomenon in Al_2O_3 during irradiation to doses much higher than the electron irradiation experiments. Figure 6 summarizes the results from published RIED studies on single crystal Al_2O_3.[49] Based on the recent ion and neutron irradiation studies, RIED does not appear to be a significant issue for doses up to at least ~1 dpa in Al_2O_3. However, surface leakage currents (and radiation-enhanced deposition of conductive surface contaminants)[51] must be accounted for in component designs since these leakage currents can greatly exceed the bulk currents in insulators.

The dielectrics which are used as windows and feedthroughs for radio-frequency heating systems in fusion reactors should have dielectric loss factors (product of the dielectric constant and loss tangent) below about 10^{-2} for ion cyclotron (~100 MHz) heating systems and $<10^{-5}$ for electron cyclotron (~100 GHz) heating systems in order to avoid excessive heating in the dielectric. Significant advances in the understanding of the effects of impurities on the frequency-dependent loss tangent of Al_2O_3 have recently been obtained.[52] Neutron irradiation can cause significant increases in the loss tangent

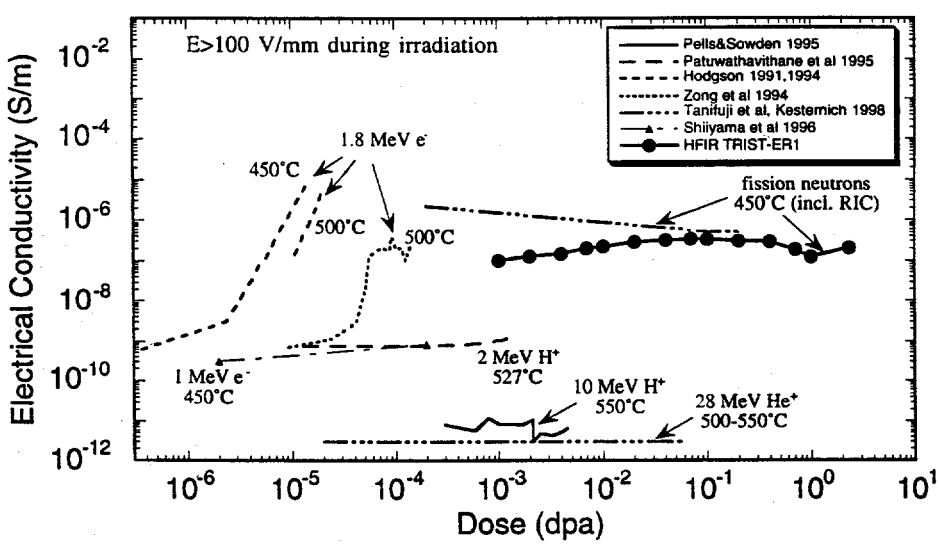

Fig. 6. Summary of RIED studies on sapphire at 450-550°C.[49] The electrical conductivities were measured at the irradiation temperature with the radiation source turned off, except for the fission reactor studies.

of dielectrics, particularly at low temperatures.[46,53] However, several materials including sapphire and CVD diamond appear to be suitable for electron cyclotron systems in fusion reactors.[53] Data on radiation-induced thermal conductivity degradation of dielectrics are reviewed elsewhere.[3,40] Significant decreases in thermal conductivity (factors of two or more) can occur during irradiation to modest doses (~1 dpa). The amount of degradation generally increases with decreasing irradiation temperature.

Very little is known about the migration enthalpies for self interstitial atoms in ceramics.[54] Recently, the rate-limiting (slowest) interstitial migration enthalpies were determined to be ~0.21 eV in $MgAl_2O_4$ and ~0.6 eV in Al_2O_3 from an analysis of the dislocation loop denuded zone widths adjacent to grain boundaries and sintering pores in ion irradiated specimens.[41] These values help to explain the superior resistance of $MgAl_2O_4$ to radiation-induced amorphization and defect cluster formation compared to Al_2O_3 at temperatures above 80 K. Amorphization generally does not occur at temperatures were point defects are mobile due to annealing effects. Similarly, the dislocation loop nucleation rate decreases rapidly with increasing defect mobility.

Recent ion irradiation studies[55,56] have indicated that significant ionization-induced point defect diffusion occurs in $MgAl_2O_4$ and Al_2O_3 for irradiation conditions where the ratio of electron-hole pair production per dpa exceeds ~50 and ~10^4, respectively. Ionization-induced diffusion (IID) effects must be considered in the data analysis for irradiated nonmetals, particularly for irradiation sources with high ionization/dpa ratios such as electrons. In some cases (e.g., MgO and Si),[55,56] athermal point defect migration appears to be possible during exposure to ionizing radiation. Many of the experimental studies on point defect production and migration in ceramics have failed to properly consider IID effects in their data analysis, and therefore need to be reanalyzed.[55,56]

A final example of an intriguing radiation effect in ceramics concerns the effect of swift heavy ion irradiation.[57] Recent work on $MgAl_2O_4$ has determined that defect cluster formation and disordering of both the cation and anion sublattices occurs in the ion tracks when the electronic stopping power exceeds a critical value of ~7.5 keV/nm.[58,59] At progressively higher doses, formation of metastable crystalline phase and then amorphization is observed. Therefore, ionization can either inhibit (via IID effects) or induce defect cluster formation, depending on the magnitude of the ionization stopping power. For $MgAl_2O_4$, IID effects have been observed for stopping powers up to at least ~3 keV/nm and defect production occurs for stopping powers above ~7.5 keV/nm.

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