Development and potential of composite moderators for elevated temperature nuclear applications

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ABSTRACT
Neutron moderators, such as nuclear-grade graphite, are essential components of high-temperature nuclear reactors. Their primary functions are to degrade the energy of fission-born neutrons into an energy range that promotes further fission and to serve as the central core structure. This paper will discuss historic reactor moderator materials, which are relatively simple monolithic materials each having intrinsic benefits and limitations. Additionally, a new class of engineered composite moderators is presented for which two examples are fabricated: magnesia-matrix composite systems with either beryllium-based or metal-hydride entrained phases. In additions to presenting their route to fabrication effectiveness as advanced moderators is discussed.

1. Introduction
The study and development of neutron moderators have been central to the realization of fission power from the initial work on purification of the Chicago Pile graphite. Since that early work a wide range of reactor types, generally classified by coolant, fuel, and moderator have been developed by the international community. Recently, compact thermal fission reactors are of increased interest due to their potential for lower and controlled construction costs, enhanced safety, and portability into remote areas. Moreover, they are considered as point-sources for process industrial heat. The compact nature of these cores by definition requires good neutron economy mandating the use of a range of material solutions with low neutron scavenging and high neutron moderation. Such core materials and engineering solutions need to be consistent with long core life and high thermal efficiency to maximize plant economics.

Optimization of compact fission reactors, within limits imposed by the use of low-enriched uranium, is driven by the interplay between that fuel, core moderator, and reflector materials. Historically, core moderator and reflector materials consist of relatively simple compounds of a single material type, with the physics of scattering favoring the lighter of the low neutron absorbing elements: H$_2$O, D$_2$O (heavy water), Be, BeO, C (graphite), ZrH$_2$, etc. The very high density of hydrogen isotopes in water and heavy water makes for the most compact thermal reactor systems. However, such systems have ultimate plant thermal efficiency limitations and safety concerns associated with loss-of-coolant events. The remainder of this paper will focus on solid moderators, and improving upon them, as enabling materials for higher temperature (more efficient), and potentially inherently safe nuclear systems. Such systems, examples being gas-cooled or fluoride-salt cooled systems, typically utilizing nuclear-grade graphite as their moderator, have moderator operating temperature in the range of 300–900°C, with some Generation IV reactors pushing that temperature somewhat above 1000°C. Moreover, these systems require their moderators to undergo fast-neutron damage which can have a profound effect on moderator thermophysical properties, almost always bad and potentially life-limiting. The typical neutron irradiation-induced dose (damage) to moderators depends on the specific reactor and location within the reactor, though can range from 1 displacement per atom to greater than 30 dpa. Operating at this upper damage range is beyond the useful lifetime of current solid moderator materials, thus mandating change-out of materials.

2. Materials performance of historic moderators and advanced moderators
Understanding the importance and then developing the technology necessary to purify Chicago-Pile grade graphite was a major step forward in the realization of
that first nuclear chain reaction. Similarly, the postulation of atomic displacement within the graphite crystal lattice by Eugene Wigner [1,2] and its implication to a range of performance properties including stored energy by Wigner and Szilard [3] anticipated the importance of irradiation damage to materials. These combined works under the Manhattan project founded the disciplines of nuclear materials and irradiation materials science. In the years and decades following the Manhattan Project, hundreds of unique nuclear reactor designs systems were envisioned and built for various applications including civilian power, naval propulsion, aerospace propulsion, and for civilian and defense purposes. The choice of neutron moderator and reflector materials were often driven by (experience,) application temperature, the dictated reactor form-factor (size, mass, etc.), and the fuel burn-up which relates to the irradiation damage of the core internals. As example, TRIGA-type research reactors operate near ambient temperature and at relatively low power density and fuel burnup. As such systems are not challenged by either temperature or irradiation damage designers opted to take advantage of the high hydrogen content of metal-hydrides, utilizing hydride-based fuel and moderator to provide a relatively compact and economic system. Another example for which compactness was a primary consideration, the Heat Transfer Reactor Experiment No. 3 (HTRE-3), a prototype nuclear aircraft engine, utilized yttrium hydride moderator and a beryllium reflector. A predecessor program, the Aircraft Reactor Experiment, utilized BeO blocks as both the moderator and reflector [4,5]. At that time, both beryllium metal and oxide were contenders, though hot-pressed beryllium oxide was selected in part due to its high-temperature performance and favorable compatibility with dry air. Its ceramic nature, i.e. high stiffness and statistical nature of fracture, was mitigated by using relatively small, segmented parts. Another program for which compact architecture was a stronger driver than fuel burnup was the Systems for Nuclear Auxiliary Power (SNAP) program with the SNAP-10A reactor tested in orbit in 1965. That system utilized a dispersion of uranium within zirconium hydride rods as combined fuel/moderator surrounded by movable beryllium reflectors. Zirconium hydride and yttrium hydride continue to be a materials of choice for space nuclear systems. For larger, terrestrial systems zirconium hydride has undergone performance testing as fast-breeder shield in the 21 MW KFK-2 Fast Breeder Reactor (FBR), with performance in canned application suggesting adequate performance for FBR and thermal power reactors [6].

In the larger military and commercial reactors graphite began and remains the moderator material of choice supporting a range of significantly varying reactor designs, defined primarily by the desired operating temperature. The continued popularity of nuclear graphite is driven both by its well understood performance in nuclear systems and because it has the best performance within the family of moderator materials which demonstrate poor stability under irradiation.

2.1. Graphite

Nuclear graphite is essentially a composite material fabricated within a nuclear specification [7], incorporating graphite coke and refined pitch products. As certain constituents are (natural) mined products and can moreover be manufactured in a number of different ways, graphite grades are unique with each possessing a range of both nonirradiated properties and as-irradiated performance. Figure 1 provides a high-level description of the processing of nuclear graphite starting with the coke product, and carrying through a series of blending, drying, forming, and graphitization steps. Nuclear graphite materials are generally distinguished by both their constituents (i.e. pitch coke or petroleum coke) and by the forming process. Historically, materials such as the Sigri Great Lakes H-451 (of the Fort St. Vrain reactor) and others of that era were extruded as large blocks (i.e. extruded graphite). These extruded forms tended to incorporate relatively large coke particles, in many cases utilizing needle coke which led to issues of pronounced anisotropic nonirradiated and irradiated properties. Moreover, the extruded graphite contained relatively large-size pores and pronounced Mrozowski-type cracks which formed during cool-down. In contrast to the extruded graphite materials, a higher degree of uniformity and finer grain structure is achieved through various molding processes with isostatic molding, a common example being the Toyo-Tanso IG-11 graphite and IG-110 nuclear graphite. Figure 1 provides a polished surface optical comparison of the GraTech International PCEA Extruded graphite and the Toyo Tanso Isomolded graphite IG-110. From the images the observable microstructure for the PCEA graphite is quite coarser with coke particles (~25 μm for IG-110 and ~ 300 μm for PCEA) and porosity approaching millimeter size, while the IG-110 material has essentially unresolvable (by optical microscopy) porosity on the order of tens of microns. The large difference in microstructure among the graphite materials can have significant impact on thermophysical properties with the isomolded graphite tending to be of higher conductivity and strength, though exhibiting fracture behavior more like a fine-ceramic: somewhat more brittle with slightly less fracture toughness [8,9]. Interestingly, while the isomolded materials exhibit a more uniform microstructure with less apparent macroscopic porosity, they in fact have somewhat lower density compared to some extruded and vibration molded graphite owing to the higher density of
very fine porosity. As example PCEA extruded graphite has a density of \( \sim 1.85 \text{ g/cc} \) as compared to 1.77 g/cc for IG-11(0). From an irradiation-damage and neutronic standpoint it is noteworthy that both materials have considerable porosity, with the isomolded graphite being nominally \( \sim 80\% \) theoretical density \( (\%_{\text{td}}) \) while extruded material is closer to \( \sim 83\%_{\text{td}} \).

The performance of graphite under irradiation is a relatively complex interplay of evolution of the graphitic constituents and the internal interfaces comprised of porosity and inherent microcracks. The most common description of irradiation-induced change assumes the anisotropic change is dimensions of the graphite crystal as the driver \([11,12]\). At relatively modest dose the effect of irradiation and its associated volumetric compression is to improve engineering properties such as strength, toughness, elastic modulus \([11,13,14]\), with thermal conductivity degradation being the important exception, as graphite conductivity is highly sensitive the excess phonon scattering created by irradiation-induced vacancies \([15–18]\). By inspection of the inset crystal of Figure 1, in the typical irradiation temperature of interest for nuclear systems of 300–1000°C neutron irradiation produces a cascade of defects, for which interstitials have the greatest mobility, forming new interstitial planes between the originally existing planes, correspondingly leaving lower mobility lattice vacancies behind. With increased irradiation, and exacerbated by increased irradiation temperature, these new planes cause a dilation in the graphite crystal perpendicular to the basal planes and a contraction within the basal plane. As the initial

**Figure 1.** Schematic of the fabrication of nuclear graphite(right) upper left, comparison of a fuel block of a prismatic and reflector block of a pebble-bed HTGR (upper left) \([10]\) and comparison of microstructure of PCEA and IG-110 nuclear graphite.
microstructural response to irradiation is to accommodate (anneal) diffusing interstitials at internal porosity, the macroscopic effect is for graphite to first degrade upon irradiation. As the internal accommodation is overwhelmed, continued anisotropic crystal swelling leads to macroscopic graphite swelling and exaggerated microcracking. Typically, the point at which the graphite returns to its initial volume (e.g. the nil swelling point) the graphite suffers significantly degraded thermophysical properties and has reached its useful lifetime. That neutron dose is dependent on temperature and to a lesser degree the nuclear graphite grade. In the lower temperature regions of a high-temperature-gas cooled reactor (HTGR, ~300–500°C) nuclear graphite blocks may last for upwards of 40 dpa, while in the elevated temperature regions (~750°C) of the core where defect mobility is much enhanced the dose to lifetime is on the order of 10 dpa [19–21]. During that course of irradiation a typical nuclear graphite will first see mechanical properties such as elastic modulus and strength increase by a more than 200 and 50%, respectively, before plummeting while the thermal conductivity may decrease by as much as an order of magnitude near 300°C and by a factor of about 2 near 1000°C [19,20,22].

2.2. Beryllium metal

Metallic Be has been considered for nuclear fuel compacts, cladding, and as fission reactor moderator dating back to the early 1950s. In addition to its favorable combination of low atomic mass and parasitic absorption, beryllium provides a neutron boost in fission power applications through its “neutron multiplier” ⁹Be (n, 2 n)⁸Be reaction. As with graphite, the Be properties strongly depend on the influence of raw materials and fabrication process on grain size and resultant impurity (oxygen: BeO) content. Properties vary significantly depending on material grade and fabrication technology [23]. While recognizing specialty optical and instrument grades of beryllium are known to provide very high quality, albeit relatively thin or small parts, for nuclear applications and in particular moderators only those processes resulting in relatively large parts are considered here.

Beryllium occurs naturally as beryll Be₂Al₂Si₂O₁₈ and chrysotberyl BeAl₂O₄. Figure 2 provides a high-level representation of how that ore is taken to final product. As depicted, once ore is reduced to the beryllium hydroxide starting material beryllium products may be classified by forming process: powder metallurgy or through melting and ingot metallurgy. As parts cast from ingot tend to have larger grains with low and nonuniform mechanical properties the powder metallurgy process is preferred, with the most common forms of Be being vacuum hot-pressed block.

Examples of current nuclear application (cf. Figure 2) of Be block include the reflector for both the High Flux Isotope Reactor and Advanced Test Reactor in the United States, and as the first wall tile for the International Thermonuclear Experimental Reactor (ITER). The most recent and most detailed work on beryllium for nuclear application has been for the ITER project, which has selected the impact ground, hot isostatic pressed S-65 C material produced by Materion and a similar grade (DshF-200) produced by the Bochvar Institute. These materials are structural grade with low (<0.1 w/o) Fe, Al, and ~1 w/% BeO, content, and guaranteed to exhibit >3% elongation (nonirradiated) at ambient.

In its pure metallic form, Be has a highly anisotropic hexagonal crystal structure with very interesting anisotropic properties. Beryllium application has been reviewed for fusion devices such as the International Thermonuclear Experimental Reactor (ITER) with high-quality materials developed, characterized, and databased [23]. A key factor limiting the use of Be, other than its toxicity, is ductility. Be mechanical behavior is known to be a function of many factors including chemical purity, grain size, testing temperature, and to some extent the testing strain rate. Fundamentally, the HCP Be crystal itself is slip resistant, severely limiting flow with the only two operating slip modes (at relevant temperatures) being basal slip on the 0001 planes and prismatic slip on then 1010 planes. The presence of oxygen (i.e. forming BeO) is well known to impact a range of properties, but most importantly ductility.

For high purity Be, ductility can be categorized into three temperature dependent regimes. On the lower end of nuclear application temperature (20–200°C) Be possesses two independent slip basal slip systems and two independent prismatic slip systems (total 4). As the Burgers vectors are normal to the basal plane at this temperature there is no c-component to slip. As the required five independent slip systems are not operative meaningful crystal plasticity is not possible leading to failure by transgranular fracture of the basal plane. Total elongation in this temperature range for high quality vacuum hot pressed material (e.g. Brush Wellman S-65 C at 150°C) is ~5% in the direction parallel to the pressing direction and ~20% in the transverse direction. As the temperature is increased into an intermediate temperature regime (~200–500°C) the critical stress for prismatic slip decreases and both slip modes combine to yield peak total elongations of about 50% both parallel and transverse to the forming direction due to anisotropic texturing of the grains. In this intermediate temperature regime the failure is primarily ductile/fibrous tearing. In the upper temperature range for nuclear application (>500°C), intergranular failure begins to occur returning the total elongation to below 20%.
Given the limitations to dislocation movement in Be, the effect of irradiation produced defects is naturally to cause further embrittlement. In the nuclear operating temperature range of interest for moderators (20 to ~800°C) the homologous temperature \( T_m \) ranges from ~19 to 68%. At the lower end of this temperature range defect mobility is very limited and it is expected that Be would be significantly embrittled by defect cluster formation. Available microstructural reporting in this temperature range [24–29] discuss high density of small dislocation loops and black spots. Early work on neutron irradiated Be has shown Stage II and III interstitial migration fully available by around room temperature [30,31] where Stage IV–V vacancy mobility is activated above 300°C, consistent with the increase in void microstructure and helium-stabilized void swelling [32–34]. In addition to defect embrittlement significant amounts of He produced through the reaction \( ^{9}\text{Be}(n,2\alpha)^2(\text{He})+2(\gamma) \) also contribute to plasticity loss [35–37]. At low temperatures He is immobile in Be, but as temperature exceeds ~500°C large He-stabilized voids form, causing excessive swelling within Be [38]. Helium stabilized bubbles can both preferentially decorate and weaken grain boundaries and form within the matrix. However, in the intermediate and high temperature regime the issue of

\[ \text{Figure 2. Schematic of beryllium fabrication routes and examples of nuclear application.} \]
helium-stabilized voids leading to substantial, non-saturated swelling which can be design limiting [39].

2.3. Beryllium oxide

Beryllium oxide ceramic is a relatively high thermal conductivity material produced through any number of powder-processing and sintering routes, not dissimilar to the powder metallurgy products approaches depicted for Be (cf. Figure 2.) Its production is typical of a “porcelain process” of powder (slurry or dry) preparation, molding, and sintering. BeO has found a range of boutique applications driven by its oxidation resistance and combined high thermal conductivity and operating temperature capability. Given the strongly covalent nature of BeO it has a relatively high melting and sintering temperature for pure powder (2570 and >1900°C, respectively). This sintering temperature can be suppressed through addition of second phase aids such as Al₂O₃ or MgO, though with significantly negative impact on properties such as thermal conductivity. Non-nuclear applications include power semiconductor devices and as structural components in high-performance microwave devices and lasers. Historically, nuclear ceramics have been fabricated from pure BeO powder or from powders which include minor additives such as MgO or (nuclear grade) ZrO₂, which has been used to stabilize a small-grained microstructure, added as powder sintering aids or a sol [40,41]. Development and study of BeO for nuclear application dates back almost as far as graphite, with BeO being the candidate reflector material (and also a core material) for the Daniel's Pile, the first conceptual design of a high-temperature gas-cooled reactor (HTGR) [40,42]. However, while considerable work BeO technology was carried out that HTGR project was ultimately canceled due to funding priorities and the aggressive nature of the technology (at that time.) Regardless, fundamental investigations into the irradiation damage in the BeO system and formation of BeO ceramics continued and the material was used widely in a number of reactor systems. As example of progress toward process, the Daniels Pile project routinely produced BeO powders of >95.5% purity and fabricated hexagonal moderator blocks of 15 cm in length and 7.6 cm across the flats. Two reactor example nuclear applications successfully deploying BeO ceramics are provided in Figure 3: (a) provides a top-down view of the Aircraft Reactor Experiment, which operated in 1954 and was comprised of pure BeO hexagonal bars (of the Daniels Reactor Project vintage) through which a 53.09 mole % NaF, 40.73 mole % ZrF₄, and 6.18 mole % UF₄ liquid fuel was passed, and (b) providing an image of the Kilopower space reactor which was tested in 2018, a solid cast 93% ²³⁵U and 7% Mo fuel surrounded by BeO reflector.

Beryllium oxide suffers from a large anisotropic dimensional change in its HCP lattice for irradiation temperatures <800°C (~40% Tₚ) due to simple defect cluster formation [43]. In this temperature range, the anisotropic dimensional changes rapidly lead to microcracking and significant mechanical degrading at less than 1 dpa. At nominally 600°C He forms on grain boundaries with generalized He bubble formation occurring above 800°C. Above ~35 Tₚ the useful temperature for fine-grained BeO (as defined by mechanical integrity/microcracking) increases substantially [44]. However, the substantial transmuted He is

Figure 3. Photographs of (a) the aircraft reactor experiment prior to reactor decommissioning and (b) the kilopower reactor under assembly.
retained in the structure, with He bubbles becomes the driver for unacceptable swelling [45]. At ~1100°C only 25% of the transmuted helium is released from the ceramic. Insufficient quality data exists on BeO to speak to the very high temperature swelling and mechanical integrity.

2.4. Metal hydrides

The ability to stably accommodate large amounts of hydrogen in metals has intriguing possibilities for nuclear applications. Metal hydrides, also known as interstitial hydrides, are typically covalently or ionically bonded and have the ability to form over a broad stoichiometric range. Two covalently bonded binary metal hydrides of interest to nuclear are YH$_{2-x}$ and ZrH$_{2-x}$, with LiH being an example of an ionically bonded candidate. The thermophysical properties of metal hydrides are comparable to or superior their metallic parents. They are reasonably good thermal conductors though markedly brittle. Relatively high values of hydrogen to metal content (H/M ratio >1) may also result from the Hydrogen-Induced Amorphization (HIA) of refractory alloys, examples being HIA’s of YFe$_2$, GdFe$_2$, etc [46]. As the H/M ratio along with the relative neutron absorption cross section of the metal is key to material’s nuclear performance the stability of the hydrogen as a function of

![Figure 4](image-url). Plateau pressures of hydrogen of various metal hydrides (after Vetrano [48]).
temperature is critical. Hydrogen retention at elevated temperatures requires specific attention to develop hydrogen barriers for the metal hydride.

Figure 4 provides an overview of hydrogen containing materials of interest and in particular plateau pressure (the partial pressure of hydrogen above a two-phase metal-hydrogen solid) for a number of binary metal hydrides. Also provided with Figure 4 is the specific hydrogen density of each material in atoms of hydrogen per cm$^3$, which can be compared with that of water ($6.6\times10^{22}$ H/cm$^3$).

Fabrication of the transition metal hydrides such as YH$_{2.4}$ and ZrH$_{2.4}$ can be accomplished through a number of routes. However, (direct or-) massive hydriding and sintering of base hydride powder are the two most common routes, each with benefits and drawbacks dependent on the application of interest. The term massive hydriding was coined to differentiate this process from the classic sintering approach which has defined geometric part size limits based on the tooling utilized (i.e. sintering die.) While typical sintering geometries are on the order of a few to tens of cm in any dimension, massively hydried parts can be substantially larger, limited by the size of the reaction chamber. In the early development of YH$_{2.4}$ for space reactor application typical massively hydride parts were 30–40 kg with dimension greater than 100 cm [47]. In massive hydriding the exothermic hydriding reaction is taken advantage of through typically slow and controlled reaction with hydrogen to avoid part cracking.

Fabrication of metal hydride through direct hydriding can be done in a variety of ways: such as introducing a measured amount of hydrogen into retort or carefully matching retort temperature and hydrogen pressure. Massive metal hydride fabrication is usually achieved using the latter method. The M-H binary phase diagram and the pressure-composition-temperature (PCT) map are the two most important pieces of information needed to guide fabrication of metal hydride. Hydriding is a rate-controlled exothermic process. The rate limiting process may be diffusion of hydrogen inside the solid metal or hydride, as H is supplied to the solid or surface adsorption, depending on the bulk H$_2$ gas flux onto the surface. The most significant challenge in fabricating massive crack-free bulk metal hydride (or during rapid dehydriding) is that if the hydrogen concentration gradient across the sample body is not managed to a low value, then differential lattice strain results lead to significant stress. This stress, coupled with the low fracture toughness of the hydride materials, is the ultimate source of cracking and it is routinely exploited to produce powder from various metals in the hydride-dehydride process. Slow hydrogen flow rate coordinated with the high processing temperature yields the crack-free metal hydride. Matching the retort temperature and hydrogen partial pressure informed with the well-established PCT map gives rise to the desired hydrogen concentration in as-fabricated hydride. The needed shape and dimension of the hydride pieces may be achieved without additional machining if the starting metals are carefully designed based on the expected volume expansion in the hydriding process.

Large-scale metal hydride may also be fabricated via the powder sintering route, starting with the high purity hydride powder. It is noted that the sintering process needs to be in the low temperature regime to avoid significant hydrogen release, altering the H/M ratio. In addition, the sintering process is cumbersome, as it involves multiple handling and processing steps, requires handling metal hydride powder in inert glove boxes, for which care is required to avoid introducing impurities in the hydride. Typically, massive hydriding allows fabrication of more complex geometries as compared to the sintered product.

Examples of direct hydride articles are provided in the upper image Figure 5, with an early and famous example of hydride, the SNAP-8 space reactor [49] provided in the lower image. Neutron irradiation response of metal hydride has not been significantly. In 1960s, the hydride Y-5%Cr alloys cladded with Fr-20Cr-4.5Al was irradiated in a nuclear reactor environment at 700°C and 1000°C for 1950 and 2450 hours in air, respectively. All irradiated specimens remain intact without weight or dimension changes. Hydrogen concentration change was also seen to be negligible [50]. Besides this study, there is no additional literature data on the neutron irradiation impact on the thermomechanical properties of yttrium hydride. As to zirconium hydride, the growth of U-ZrH fuel during irradiation has been studied extensively in the SNAP reactor program. Dimensional change is dependent on the burnup, ranging from 0.6% to 1.4% at 760°C [51]. Some other irradiation experiments indicate negligible change in the density of ZrH$_{1.7}$ after irradiation to fluences of $7 \times 10^{21}$ to $1.1 \times 10^{22}$ n/cm$^2$ ($E > 1$ MeV, just under 1 dpa) at temperatures up to 570°C [52].

3. Development of composite moderator systems

Moving beyond the historic moderators discussed in the previous section, engineered materials are an option for moderator and reflector application. A number of example composite materials and advanced manufacturing techniques may be considered aligned along certain principles including the need for acceptable neutronics, environmental stability, fabricability, and economic viability of the material system as applied to design. One conceptually simple system is a multiphase composite, where a highly moderating entrained phase is contained within a continuous and environmentally stable matrix
phase. The conceptual architecture for such a composite, and in particular the desired attributes for a composite moderator are provided in Figure 6. Such a random or ordered two-phase composite is one of many types of composite forms which could be envisioned, some of which could be designed to simultaneously enhance engineering properties of interest such as thermal conductivity (as example, through nonuniform incorporation of high-conductivity fibers) or enhancing strength and fracture toughness (as

Figure 5. (a) Photographs of as-fabricated yttrium hydride and zirconium hydride through direct hydriding. (b) A systems nuclear auxiliary power (SNAP)-8 nuclear reactor core, showing some of the cylindrical U-ZrHx fuel elements, clad in a nickel-steel superalloy. The core is approximately 20 cm in diameter [49].
3.1. **Entrained phases of a two-phase composite moderator**

As the in service thermal and mechanical property requirement of an entrained phase are relatively unimportant (see Figure 6) material selection is relatively straightforward focusing on materials processing issues, benefit to overall moderation with minimal neutron scavenging, and susceptibility to environmental degradation. In this application environment effects include long-term thermal stability under irradiation and the potential interaction of the entrained phase with the host matrix. An abbreviated list of candidate entrained phases are provided in Table 1 below, focusing on thermally stable compounds of high density, low-atomic numbered constituents. As neutron moderation is physically described by simple elastic “billiard-ball-type” collision, the most efficient entrained phase moderators possess high densities of elements with mass approaching that of a neutron: H, Li, Be, B, C ... Along with the logarithmic energy decrement $\xi$, the atomic mass and number density of elements in a material determined the **slowing down power**, a primary metric for comparing the effectiveness of materials as moderators: Slowing Down Power = $\xi N_d \sigma$, where $N_d$ is the atomic number density and $\sigma$ is the cross-section for neutron scattering. In Table 1 and Figure 7 the slowing down power of potential entrained phases are compared to the two most common moderators, graphite, and water. As seen, the slowing down power for water far exceeds that of graphite, owing the very high density of low atomic number hydrogen. For this reason, light-water-reactors (LWR’s) enable relatively small and very high-power density cores, especially as compared to the higher temperature graphite-moderator gas-cooled systems for which the materials in this work are being developed for.

Beyond simple moderation, neutron scavenging becomes very important, with the thermal neutron absorption ($\sigma_n$) of constituent elements provided in Table 1. This is typically quantified as the Moderating Ratio: $\xi N_d \sigma_n/\sigma$. For these metrics of neutron scattering and absorption it is clear why the historic moderators graphite, beryllium, and beryllium oxide were chosen. Each is of high scattering due to its high density of light atoms and each possesses a relatively
neutron absorption cross section. Moreover, the naturally occurring unstable isotopes of these elements all possess low absorption. This is in contrast with a number of materials of interest for which naturally occurring isotopes can have vastly different neutron absorption cross sections. The classic example of this is boron, which has an average neutron absorption cross-section of 767 barns (1 barn = 1×10^{-24}/cm²), though the absorption of the two naturally occurring are substantially different: $^{10}$B isotope having
a naturally occurring abundance of 20% and a cross section of 3835 barns while the majority (80%) $^{11}\text{B}$ isotope has a relatively small cross section of 0.0055 barns. This is captured in the table with the primary isotope and average isotope cross sections listed separately. Another example important to the nuclear industry are the isotopes of hydrogen, with $^1\text{H}$ having a neutron absorption of 0.333 barns in comparison to the much reduced 0.0005 of $^2\text{D}$ (deuterium.) This disparity is why heavy water was (along with purified graphite) the two original moderating materials utilized to determine the possibility of the fission chain reaction. For the purposes of Table 1 and in discussion of potential entrained phases for moderating composites it is important to note that isotopic tailoring, or the separation and use of certain low absorbing isotopes, is a known technology, albeit potentially costly. As example, deuterium is commonly separated for use in heavy water reactors and $^{10}\text{B}$ isotope is routinely separated from natural boron for nuclear application leaving the reduced cross-section $^{11}\text{B}$ isotope behind as by-product.

While technically feasible, and noting the relatively attractive slowing down power of the beryllium borides of Table 1, these materials are not pursued here essentially because the potential cost impact of isotopic tailoring. However, a number of materials in the B-Be family have attractive slowing down powers, and absent $^{10}\text{B}$, have attractive moderating radio. While the first identified phase of beryllium boride was made by Lebeau in 1896 [54] the earliest report discussing the processing of the Be-B system with corresponding structure identification was carried out by Markovskii et al in 1955 [55]. There, reaction was carried out Be-B reaction in a range of 20:1 to 1:6 in vacuum and hydrogen from 1300 to 1400°C, with identification of BeB$_6$. As has been made apparent since that time, in the Be-B family a range of materials occurs, from Be-rich to B-rich phases with some stable at the intermediate to high temperature considered here. However, the only Be-rich phase that appears to be stable at room temperature is Be$_4$B. On the boron-rich side a BeB$_3$, BeB$_4$, BeB$_5$, and BeB$_6$ have been described [56–61], though study has been somewhat limited and in fact controversy still exists as to whether BeB$_3$ exists, or is in fact a more complex structure such as BeB$_2$[57]. These beryllium borides have never been studied in a serious way for nuclear application, with interests being either purely scientific or in support of light armor or other lightweight, high stiffness application. Currently, there exists no data on irradiation stability of this family of materials with little information on its reaction with water or air at elevated temperature. However, it would be assumed that formation of beryllia would provide some level of protection.

Like beryllium-boron, Lebeau is also credited for the discovery and early work on beryllium carbide, Be$_2$C [62,63]. Unlike the beryllium-boron system, Be$_2$C with its cubic structure is the only phase that exists in the Be-C system. It is both a very hard and abrasive material with density as determined through bulk density measurement [64,65] and X-ray [66,67] of 2.44 g/cc, though it is noted that in early work on the mass production of Be$_2$C [68] a much reduced density was reported, likely due to inclusions of graphite and high levels of BeO present in the compacts. This lower, incorrect density has unfortunately been propagated through online resources such as MatWeb, Wikipedia, and the Compendium of Material Composition Data for Radiation Transport Modeling [69].

In Lebeau’s original work two generic methods of forming Be$_2$C were the reaction of Be metal or reduction of BeO with “sugar charcoal.” While the reaction of BeO at high temperature has been pursued as a potential more commercial process [64,68] the formation and sintering of pure powders appears to result in higher quality product. It is noted that direct reaction of Be metal with graphite powder, which reacts exothermically upon reaching 900°C, is another viable option. As Be$_2$C reacts with moist air progressively hydrolyzing to form Be(OH)$_2$ consolidation is best carried out in inert environments. A number of standard, dry, powder ceramic techniques are possible and result in fully dense ceramic [64,65]. In the bulk Be$_2$C is not attacked by moist air, but due to its significant reaction with nitrogen above 1100°C and by oxygen above 800°C it has not receive commercial application. It is noted that these environmental conditions may not be of concern in certain moderator application. Finally, the irradiation stability of Be$_2$C is essentially unknown, with the only known study carried out below 90°C to a fluence of less than 0.01 dpa (3.5 x 10$^{22}$ n/m$^2$.) [70] Little physical property change was noted at this very low fluence.

Beryllium intermetallic compounds (beryllides), examples listed in Table 1 being ZrBe$_{13}$, TiBe$_{12}$, and VBe$_{12}$, have received sporadic interest from the fundamental materials science, nuclear fission, aerospace, and nuclear fusion communities over several decades [71–78]. Apart from their exceptional high temperature mechanical properties, the relatively high Be number density gives them very attractive moderating potential. In particular, the slowing down power of ZrBe$_{13}$ falls between that of Be metal and BeO. While all the beryllides will have a lower moderating ratio than Be or BeO given the higher thermal neutron absorption cross section of the metal (Zr, Ti, or V), the ZrBe$_{13}$ in particular possesses a relatively low neutronic penalty as compared to BeO. Potential advantages of the intermetallic systems as compared to BeO are their higher as-irradiated thermal conductivity and overall irradiation stability. However, as a family the understanding
of the irradiation stability is poorly studied, with only limited information on the TiBe$_{12}$ available [79,80]. In the recent work of Fedorov [80] in the 145°C-725°C range and doses to $1.4 \times 10^{26}$ n/m$^2$ (E $> 0.1$ MeV) (a dose and temperature range of relevance for moderator application), materials exhibited a few percent isotropic swelling and phase stability. As comparison, at that dose and temperature the anisotropic dimensional change of BeO would have led to severe microcracking and loss of strength and Be metal would incur unacceptably high swelling.

Through entrainment in a dense composite matrix which would inhibit decomposition, hydrides offer tremendous potential to significantly enhance neutron moderation. As discussed in Section 2, the technology of forming monolithic metal hydride parts of high quality has been established and a technology readiness level established for their use in reactor. However, safety and performance limitation with regards to thermal and irradiation-induced loss of hydrogen. Referring to Figure 4 a large number of metal hydrides of sufficient thermal stability to be considered as composite entrainment materials, depending ultimately on the processing conditions. Table 1 provides a list narrowed by considering the absorption cross section of the metal. As seen, the hydrogen density is relatively close for the selected metal hydrides with ThH$_2$ being at the upper end of the spectrum. However, while ThH$_2$ is often considered for nuclear application the relatively large absorption cross-section argues against its use.

### 3.2. Matrix phases of a two-phase composite moderator

Referring again to the composite schematic of Figure 6, the fundamental difference when selecting a matrix material for a two-phase moderator as compared to the entrained phase is the additional thermo-mechanical performance requirements and in some cases the need to retain hydrogen. As envisioned, the matrix phase would be the primary load-carrying constituent of the composite, where the primary load existing in classic graphite stacked-block gas-cooled reactors is compressive, with both fracture toughness and tensile strength becoming important as the structure evolves under irradiation-induced dimensional change. As such stresses are compounded by thermal gradients the desire for higher conductivity (as irradiated) is also of importance. As mentioned in the introduction the application temperature range of interest can range within a typical graphite moderated gas-cooled reactor from 300 to 900°C or higher. This temperature, combined with the need for very low neutron absorption (typically $\sigma < 0.1$ barn for a low-enriched-uranium thermal spectrum reactor) defines the potential matrix materials.

Metal-matrix, intermetallic-matrix, and ceramic-matrix composites may be considered as advanced moderators. However, within the limitation of temperature and neutron absorption metal-matrix candidates are extremely limited. Be-metal is conceivable, though for the upper-end of the desired temperature and dose regime irradiation swelling and embrittlement are significant issues. Other, low neutron absorbing metal such as Al and Mg soften and melt within the desired application temperature range, could be considered at low temperatures. Intermetallic matrices offer the potential for higher temperature application, though of the most commonly developed alloys none is neutronically acceptable. Less well-developed intermetallic systems, such as the select beryllium intermetallics such as ZrB$_{12}$ (cf Table 1) and potentially materials in the Mg- and Ca-based systems have potential.

Ceramic matrices appear to offer the most potential for high-temperature composite moderator application. Many offer reasonable neutron moderation as well as acceptable neutron absorption and high-temperature performance. The most well studied example is SiC, which can be matrix-processed using a wide range of methods, though from an irradiation stability point of view [81–84], matrices processed either by chemical vapor infiltration (CVI/CVD) or through a transient-eutectic sintering process appear the most promising. While processing temperature to achieve a full density matrix can be below 1800°C [85,86], the neutron scavenging of Si (cf Table 1) renders SiC less than attractive as a moderator. Higher thermal conductivity ceramics such as Si$_3$N$_4$ and AlN can be similarly excluded. Glass shares the same issue of Si neutron absorption but should also be ruled out based on the fundamental irradiation-instability of the structure [87,88]. However, glass ceramics in the ZAS (ZnO $\times$ Al$_2$O$_3$ $\times$ nSiO$_2$), LAS (Li$_2$O $\times$ Al$_2$O$_3$ $\times$ nSiO$_2$), and MAS (MgO $\times$ Al$_2$O$_3$ $\times$ nSiO$_2$) assuming marginal neutron scavenging can be tolerated and isotopic tailoring for the case of the lithium. It is noted that known compositions do include glass ceramics with reduced Al and Si concentration. (For example, GOX glass ceramic based on parent glass composition 39.5SiO$_2$ $\times$125Al$_2$O$_3$-21.5CaO-12.5 MgO-12.5YO$_2$-1.5ZrO$_2$) [89]

Limited data on irradiation performance of glass ceramics suggest a level of stability [90].

Stabilized ZrO$_2$ and Al$_2$O$_3$ are indifferent performers neutronically, though both have excellent high-temperature stability in a range of environments with Al$_2$O$_3$ being relatively stable under irradiation [45,91] and ZrO$_2$ less so [92,93]. However, the high coefficient of thermal expansion ($11 \times 10^{-6}$/K) of ZrO$_2$ combined with it extremely low thermal conductivity makes it a poor choice as a block moderator material. The conventional sintering of polycrystalline pure Al$_2$O$_3$ is
a grain boundary diffusion limited process beginning at approximately 1400°C and reaching near full density at around 1700°C [94]. Various approaches in powder processing including high-purity and finer powder, sintering aids, and the application of spark plasma sintering can be used to reduce the temperature at which a dense compact is achieved by a few hundred degrees Celsius [95,96]. Techniques such as spark plasma sintering of pure alumina powder and powder including sintering aids demonstrate dense ceramic at 1300°C [97,98].

Given the reasonable scattering and extraordinarily low neutron absorption of Mg, the family of refractory oxides containing magnesium are of particular interest. MgAl₂O₄ or Magnesium Aluminate spinel (MgA) while a relatively weak ceramic, is known for its high-temperature use and irradiation stability, being widely used as refractory brick in kilns, ladle lining in steelmaking, and having been variously studied as a fuel matrix and waste form material [45,91,99–101]. As there is limited natural occurring MA the ceramic is typically formed through solid state reaction of MA-bearing precursors such as Al₂O₃ and MgO, which are readily available. Direct sintering of commercially produced MA powder is common, through the usual processes of hot-pressing, hot-isostatic pressing, spark plasma sintering, etc. though given the availability and purity of Al₂O₃ and MgO, the precursor powder route is preferred [102]. Importantly, a significant volume change (~8%) occurs during MA formation, leading to a usual dual stage sintering in the 1000–1600°C range which increases cost.

Due to the presence of Al the overall absorption of MgAl₂O₄ may not be ideal for some moderator application. In contrast, magnesia (MgO) is a superior neutronic candidate as both elements Mg and O (cf Table 1) have very low cross sections and the overall slowing down power is ~5% that of graphite, providing the composite a moderating boost from the matrix to compliment the entrained phase. To this point there has been little discussion of MgO as a bulk moderator [103] though it has been discussed as both inert matrix for nuclear fuel [104–106] and as a waste form barrier material [107]. MgO ceramics have widespread commercial application, as a primary crucible material and a number of other common uses such as construction flame resistant fireboard. It is one of the most studied refractory ceramics with an extensive database with example applications dating to the Great Wall of China and early Roman construction. Final form materials vary widely, including fibers, porous structures, composite, and sintered bodies of varied density. For discussion purposes and related to reactor moderator application materials and processes for densities in excess of 98% are desired.

Sintering of pure MgO powder into dense ceramic is readily achieved at temperatures in the sintering range of 1200–1650°C, with lower temperatures achieved through application of pressure and by moving toward smaller and purer powders including nanopowder. As example >99% theoretical density can be achieved, with restrained grain growth, by application of relatively high pressure (170MPa) and by using nanopowder [108] at 1470°C. Use of spark plasma sintering enables grain growth control, providing a more thermal shock resistant ceramic, by inhibiting grain growth through a rapid heating stage leading to final sintering [108]. Further suppression in sintering temperature can be achieved through the use of elemental sintering aids such as Al, Cr, Fe, Mn, V, lithium-bearing salts [109] and various oxides [110]. Compounds such as Li-bearing salts [111,112] and oxides such as TiO₂ [110] have also been shown to produce relatively low-temperature full-density ceramic [109,113]. With regards to irradiation stability the database for dense MgO ceramics is not comprehensive, though existing data [91,100] suggest good dimensional stability under irradiation in the temperature range of interest to moderator (300–900°C). As example Clinard [91], in comparing the irradiation swelling of Al₂O₃ with MgO find MgO to swell to ~ 3% by 30 dpa while alumina exhibits ~6% swelling. This can be compared with nuclear graphite at a similar dose and temperature, which would have initially densified in excess of 5% and then reversed to swelling behavior and catastrophic loss of mechanical properties [20]. Moreover, due to the cubic (Fm3m rocksalt) crystal structure of MgO the lattice undergoes isotropic expansion as compared to the undesirable anisotropic expansion and related stress-induced reduction in strength of graphite, BeO, and Al₂O₃.

### 3.3. Neutronic impact of proposed two-phase composite moderators

In the following sections demonstrations of two-phase moderators, composed of materials discussed in the preceding sections, are described in some detail. However, prior to that materials description it is instructive to demonstrate the potential neutronic performance for these systems. Table 1 provides insight into both slowing down power and moderating ratio, perhaps the two most important metrics for a moderating material. However, as the materials being discussed are essentially neutronically tunable through selection and volumetric fraction of their constituents and in many cases, it is possible to over moderate a system, it is ultimately important to engineer one or more moderators for each specific reactor or reactor type. Regardless, the most simplistic description of the effectiveness of the suggested composite moderator is made by determining the critical volume of a sphere, where the fuel is homogeneously dispersed within the moderator and the sphere size required to reach “reactor” criticality is estimated. This
is provided in Figure 7. In this simple example a number of selected MgO-matrix composites result in significantly smaller critical volumes as compared to nuclear graphite, with the MgO-15% ZrH$_{1.6}$ have under half the required volume. The MgO-matrix composite entrained with either Be or BeO also show significant benefit, while the SiC-matrix, due to the greater neutron absorption of the Si atom, has poor performance. For similar reasons composite moderator fabricated from MgAlO$_4$ and Al$_2$O$_3$ are less attractive than the MgO-based moderators.

Clearly, the composite moderators have a potentially large and beneficial impact on core size and associated uranium loading, though the enhanced moderation can lead to serious consequences on reactivity coefficients and other important safety and performance parameters and must be carefully examined. Such work has been carried out (REF including thesis?) whereby composite moderators are considered as direct replacement into a well-studied modular high-temperature gas-cooled reactor (mHTGR) configuration previously optimized for nuclear graphite. In that detailed work, assembly level calculations were performed for composite moderators to evaluate performance and compared against a graphite moderated system. The analysis showed that the composite moderators could provide similar or enhanced cycle performance relative to graphite. The improved cycle performance results in lower fuel cycle costs and a lower environmental impact for the composite moderators relative to graphite. The composite moderators also have a softer neutron spectrum which leads to less radioactivity per unit energy generated which is expected to reduce the shielding and long-term storage requirements of the spent nuclear fuel. Calculations of critical core size were performed for a hypothetical nonreflected homogenous spherical reactor showed that the most promising composite moderators, that consisted of a MgO-matrix composite entrained with either hydride, Be, or BeO, provided a smaller size than for graphite moderated system, also illustrated in Figure 8.

Thermal-hydraulic safety calculations were also performed in the aforementioned study for the most promising beryllium-based composite moderators. The beryllium-based moderators have a lower thermal conductivity but higher volumetric heat capacity than that of graphite. Calculations showed that for accidents that result in an absences of active core cooling, no moderated system is expect to compromise the fuel. The differing thermophysical properties of the beryllium-based moderators to graphite results in a lower fuel temperature increase but longer times at elevated temperatures.

3.4. Fabrication of MgO-matrix two-phase composite moderators

3.4.1. Reduced temperature MgO processing

Magnesia has been relatively widely studied as a second (entrained) phase material additive, whether in particle or whisker form, for a number of ceramic

![Figure 8. Core critical volume of a nonreflected sphere of various composite moderators of optimized fuel/moderator ratio including a uniform distribution of TRISO fuel (e.g. UCO, enrichment = 15%).](image-url)
and alloy-matrix composites. However, there has been little interest in dense MgO as composite matrix, likely due to the relatively low strength and fracture toughness of the ceramic. Some published works explore the MgO-Al₂O₃ (a spinel precursor) [114,115] system, MgO as matrix for inert-matrix nuclear fuels [104], and as composite with SiC reinforcement in fundamental oxidation studies [116]. As discussed in Section 3.2 dense magnesia ceramic is achieved through a wide array of high-temperature sintering techniques, with a number of processing routes and formulations effective to reduce sintering temperature and controlling grain size. Of primary interest for MgO-matrix moderators is the achievement of a high-density matrix processed below any reaction or melt/disassociation limits of the entrained phase. Reduction of sintering temperature, time, and pressure is therefore of interest.

For the present work sintering of powder with a single or bi-modal particle size distribution was carried out using direct current sintering, schematically shown in Figure 9. For single particle size distribution an 99.9% purity powder of < 5 microns is effective. Sintering temperature was suppressed through appropriate selection of size and purity of MgO powder and the use of a lithium-bearing salt additive (>99.9% pure) at 1%. Salt-bearing powders pre-mixed and cold pressed a nominal sintering applied pressure of 10 MPa resulted in a much-

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**Figure 9.** Schematic describing the lithium-salt assisted direct current sintering of MgO composite with entrained phases of Be metal, BeO, Be₂C, or ZrHₓ (or YHₓ).
reduced temperature (cf Figure 9) for both onset of sintering and temperature required to achieve full (>98% theoretical MgO) density. Specifically, at the 1% additive level LiCl, LiBr and LiF reduced the sintering onset temperature from ~1070°C to ~890°C (LiCl), ~860°C (LiBr), and ~740°C (LiF) [117,118]. Full density (>98% theoretical) was achieved at 1030°C for the MgO-1% LiF and 1200°C for the MgO-1%LiBr. At the upper sintering temperature of 1200°C the LiCl and pure MgO achieved ~97% theoretical density. From Figure 9 it is noted that for the MgO-1% LiF there is a weak dependence of final density on applied pressure, with the majority of densification occurring by 850°C. For this reason, and for the remaining discussion in this paper, 10 MPa applied pressure is assumed.

Beyond the demonstrated efficacy of the Li-bearing salt in suppressing sintering temperature, selection of this sintering route has a clear advantage insofar as the salt is a fugitive sintering aid: according to previous work [119] in the Al₂O₃-LiF system it would be expected that LiF evaporates from the system at approximately 845°C. This was demonstrated through a series of increasing temperature sintering studies of MgO-1%LiF as shown in Figure 10. Referring to the

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**Figure 10.** Evaporation of the LiF as demonstrated through analysis of MgO-1% LiF ceramic X-ray patterns at 900°C, 1000°C, and 1100°C.
topmost scan of the figure, which provides (as blue squares) the 45° and 65.2° location of the diffraction peaks. With increasing sintering temperature both of these peaks diminish substantially with the 65° peak disappearing as a result of the 1100°C sintering. SEM/EDS analysis confirms the absence of LiF in this 10 μm grain size sintered MgO.

As discussed in section 3.2 a range of sintering aid additives have been shown effective to enhance low-temperature sintering of MgO. The physical mechanism for this impressive shift in the sintering temperature window (~350°C) is, at the present time unknown. Previous works have speculated that LiF acts as an accelerant to the sintering process, either through the formation of a reactive liquid phase that “wets” the surface of MgO grains, or through a plastic deformation process [109,111,120–122]. Based on our initial results and these past works, we speculate that the Li dopants form complex phases with the MgO enhancing surface diffusion and sintering. To test this hypothesis, we performed in situ high energy X-ray diffraction experiments at the X-ray Powder Diffraction (XPD) beamline of the National Synchrotron Light Source-II (NSLS-II) [123]. The high flux, high-energy, and high-temperature heating capabilities at the XPD beamline [124] effectively facilitate capturing the atomic and crystallographic evolution while sintering MgO and shed light on the phase evolution with the lithium bearing salt dopants.

Utilizing the NSLS-II, the time resolved X-ray capabilities (sub-second-to-second time scale) of XPD are needed to resolve the evolution of the atomic structure of the Li-MgO powder mixtures as the specimens are cycled through the sintering temperature window (RT–1100°C). In situ sintering experiments were carried out with lithium salt doped MgO: 5.0 wt.% LiF +MgO mixtures loaded into a custom hexapole lamp furnace sintering cell. XRD patterns were measured from RT to 1100°C. The raw 2D XRD images were reduced 1D XRD patterns and analyzed using high-throughput software to quantify the evolution of the lattice [125,126].

The in situ XRD patterns are shown in Figure 11(a) as a function of temperature (up to 871 K). The initial phases in the powders are LiF, MgO, and Mg(HO)2. After 572 K, the Mg(HO)2 phase decomposes. Note that a minor phase (currently unidentified) is observable at 519 K onward, with subtle peaks at two-theta 6.63 and 5.35 degrees. The change in the positions of the XRD peaks for the LiF and MgO to lower two theta positions with higher temperature is indicative of thermal expansion and/or defect formation. Figure 11(b) shows the lattice parameter for MgO determined from the quantitative Rietveld refinements as a function of temperature, with reference MgO shown as red symbols [127]. The clear deviation in the elevated temperature lattice parameters for the MgO + LiF mixture, compared to the MgO reference, indicates that a substantial higher point defect population must exist. The temperature at which the deviation occurs, ~500 K, is well below the decomposition of the Mg(HO)2 and LiF phases (572 and 1100 K, respectively), and intriguingly close to the onset of the formation of the unidentified phase. Clearly the lattice response in this temperature range is complex and below the onset of sintering. Additionally, the large difference in the lattice parameters for the MgO + LiF mixture and reference MgO persist at elevated temperatures where enhanced sintering is observable. A liquid phase sintering mechanism does not appear to be plausible, due to the significant densification at temperatures where no liquid is observed. We thus confirm our initial speculation that the fugitive LiF promotes the formation of a crystalline eutectic phase as well as a substantially high point defect concentration (even in the absence of applied pressure), which synergistically act together to enhanced sintering and grain growth in MgO. Moreover, it appears clear that the effect of the Li-bearing salt on sintering is not solely a surface phenomenon. We plan to pursue this phenomenon with future in situ XRD experiments using a wider range of lithium-salt addition and including a range of the salts to isolate the emergent phase. These in situ

![Figure 11](https://image.pollj.com/)

*Figure 11. (a) In situ XRD patterns for MgO + 5 wt.% LiF powder mixture. (b) Quantitative lattice parameters for the MgO phase as a function of temperature. Lattice parameter for bulk MgO (with no LiF sintering additive) is given for reference (data are from Ref [127]).*
experiments are appreciably sensitive to the atomic defects and crystallographic phase evolution and shed considerable light on the sintering dynamics in this technologically relevant material.

3.4.2. Example MgO-matrix two-phase moderators

In comparison with potential SiC, Al₂O₃, or MgAl₂O₄-matrix systems, the MgO-matrix system as described above allows a very wide range of potential entrained phases due to low required sintering pressure (10 MPa or less) and very low processing temperature (~730–1030°C). Referring to temperature capability of the entrainment materials Table 1 it appears that the potential exists for a MgO-matrix composite to incorporate most of the identified materials including Be-metal, the low-temperature ceramic moderators such as Be₄C and Be₂C, and remarkably, a wide array of metal hydrides. At this point a number of two-phase moderators have been produced following the generic MgO sintering recipe outlined in Figure 9 in the entrainment volume fraction range of 10–40%. Two-dimensional x-ray tomography slices through example moderating composites are provided in the images Figure 12.

For the case of the Be-metal of (Figure 12 (a)) sintering occurred normally though a limited reaction occurs at the matrix-entrainment interface. The Be-metal particle size utilized was approximately 400 μm with a purity of >99.9%. During sintering a reaction occurs, enhanced by the presence of LiF, whereby Be reduces the MgO to form BeO on the core of the powder with a limited amount of free Mg forming MgF₂, a high melting point (1263°C) tetragonal phase. Depending on initial Be-metal size a core-shell microstructure results with the entrainment being a Be-core surrounded by the BeO reaction product. While this phase can be limited through appropriate selection of sintering conditions and is not a neutronic penalty as both Mg and F have low neutron absorption cross sections, it may be undesirable given its tetragonal structure insofar as composite irradiation stability is concerned. Using BeO as starting powder in place of Be obviously eliminates this reaction, resulting in highly dense and phase-pure composite (Figure 12 (b)), with only a small penalty over the Be-metal composite. Figure 13 provides the DCS sintering curve, thermal conductivity and thermal expansion for this composite. As expected, the thermal conductivity is

Figure 12. X-Ray tomographic images of four two-phase composite moderators: (a) MgO-40%Be, (b) MgO-40%BeO, (c) MgO-30%Be₂C, and (d) MgO:20%ZH₂. All composites fabricated with a 1% LiF additive with a 10 MPa applied pressure, 50°C/min ramp rate to a flat-top hold of 10 minutes at 1100°C. Sample cooling was carried out at a nominal 50°C/min rate.
somewhat lower than the rule-of-mixtures (ROM) value (~180 W/m-K at room temperature,) though agrees well with the Maxwell model as applied to composite. Also of note is that as the BeO volume fraction of this powder is increased from 10 to 40 vol% the composite relative (to theoretic ROM) density decreases, with a large intrinsic porosity of the powder itself. In this case the porosity is an engineered solution to potential entrainment strains produced due to helium transmutation or the anisotropic swelling of the BeO. This engineered porosity can be reduced or eliminated by careful selection and treatment of the BeO. As determined by x-ray tomography the matrix MgO phase remains fully dense with an absence of large or interconnected porosity.

The formation of MgO-Be2C composite, image provided in Figure 12 (c) followed the same processing as that of the BeO composite, using Be2C powder fabricated through the reaction of nominally 3 μm graphite powder reacted with beryllium vapor. The resulting powder was highly crystalline Be2C of similar size distribution.

The final example composite is provided in 12 (d), being a MgO-ZrH1.6 composite where the hydride loading was ~20% of ZrH2 (ε phase), which thermally decomposes during processing to form the (more irradiation stable δ phase) ZrH1.6. While higher hydride-loaded composites were fabricated, neutronic analysis suggests [128] that compositions near 20% hydride are optimal for high fuel packing fractions (~0.50) so as not to over-moderate the reactor system. Figure 14 shows the calculated initial reactivity for a hydride moderated micro-HTGR. The initial reactivity of a system can provide an insight on the optimal core configuration. As shown, the hydride moderated system is very sensitive to changes in the volume fraction of the entrained moderator and fuel packing fraction. A significant processing difference between the hydride-entrained and beryllium-entrained composite is the application of hydrogen-overpressure during DCS sintering for the case of the hydride-entrained composite. Simply, by providing a partial pressure of hydrogen environment during processing the effective plateau pressure (cf

![Figure 13. Sintering curve and thermophysical properties for a MgO-40%BeO composite.](image1)

![Figure 14. Initial reactivity for a hydride moderated micro-HTGR as a function of fuel packing fraction and entrained moderator volume percent.](image2)
Figure 4) is shifted to the right, effectively increasing the allowable processing window for these materials [129].

4. Concluding remarks

As a result of the work under this US Department of Energy ARPA-E Meitner grant, a range of new composite materials have envisioned, fabricated, and analyzed for their performance as high-temperature moderators for nuclear systems. General conclusions regarding the applicability of these systems elevated temperature reactor moderators are as follows:

(1) The simple example of two phase, ceramic matrix composite based on the MgO matrix with highly moderating entrained phase offer superior neutron capability to the historic graphite they are intended to replace. Potential enabling attributes of these advanced composite moderators as compared to nuclear graphite are potentially decreased core size, decreased uranium loading, less drastic irradiated property changes, and lifetime components.

(2) Two classes of MgO-based composite moderators have been demonstrated: beryllium-based and metal hydride-based. The entrained phases, such as BeO and ZrH₁₋₅, have been incorporated into Mg the range of 10–40 volume %. For the BeO-based composite a fully dense matrix with not interconnected porosity has been realized. For the metal-hydride composite a much lower volume of hydride (15–20 volume %) is anticipated as most appropriate for application to avoid system overmoderation.

(3) As this class of moderating composite is new to nuclear materials, there is currently a range of important performance information needed such as cost, detailed core neutronic analysis including impact on safety, performance under irradiation, and off-normal environmental attack, such as air ingress etc.

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