BNL-112576-2016-JA

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Submitted to JOURNAL OF NUCLEAR MATERIALS

August 2016

Nuclear Sciences & Technology Department

Brookhaven National Laboratory

**U.S. Department of Energy
USDOE Office of Science (SC),
Nuclear Physics (NP) (SC-26)**

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PROTON IRRADIATION EFFECTS ON BERYLLIUM – A MACROSCOPIC ASSESSMENT

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ABSTRACT:

Beryllium, due to its excellent neutron multiplication and moderation properties, in conjunction with its good thermal properties, is under consideration for use as plasma facing material in fusion reactors and as a very effective neutron reflector in fission reactors. While it is characterized by unique combination of structural, chemical, atomic number, and neutron absorption cross section it suffers, however, from irradiation generated transmutation gases such as helium and tritium which exhibit low solubility leading to supersaturation of the Be matrix and tend to precipitate into bubbles that coalesce and induce swelling and embrittlement thus degrading the metal and limiting its lifetime. Utilization of beryllium as a pion production low-Z target in high power proton accelerators has been sought both for its low Z and good thermal properties in an effort to mitigate thermos-mechanical shock that is expected to be induced under the multi-MW power demand.

To assess irradiation-induced changes in the thermal and mechanical properties of Beryllium, a study focusing on proton irradiation damage effects has been undertaken using 200 MeV protons from the Brookhaven National Laboratory Linac and followed by a multi-faceted post-irradiation analysis that included the thermal and volumetric stability of irradiated beryllium, the stress-strain behavior and its ductility loss as a function of proton fluence and the effects of proton irradiation on the microstructure using synchrotron X-ray diffraction. The mimicking of high temperature irradiation of Beryllium via high temperature annealing schemes has been conducted as part of the post-irradiation study. This paper focuses on the thermal stability and mechanical property changes of the proton irradiated beryllium **and** presents results of the macroscopic property changes of Beryllium deduced from thermal and mechanical tests.

KEYWORDS: *Irradiation damage, thermal cycling, annealing, beryllium*

* Work performed under the auspices of the US DOE

Introduction

Beryllium, exhibiting excellent neutron multiplication and moderation properties in conjunction with its good thermal properties, is under consideration for use as plasma facing material in fusion reactors and as a very effective neutron reflector in fission reactors [1, 2]. While its relatively low thermal neutron absorption cross section makes beryllium the material of choice for reactor reflectors, long term exposure to neutrons may result in gradual buildup of transmutation gases such as helium and hydrogen the buildup of which is linked to swelling and degradation of its mechanical properties. The irradiation generated transmutation gas products exhibit low solubility leading to supersaturation of the beryllium matrix and to eventual precipitation into bubbles that coalesce resulting in swelling and embrittlement, degradation of the metal properties and the limiting of its lifetime [3-6].

In addition to its consideration as plasma facing material in fusion reactors and as reflector in fission reactors beryllium has been also considered in hybrid spallation neutron source targets or as moderators/reflectors and as primary pion production target in a number of high power accelerator initiatives [7-13] where low-Z target material is sought for a desired pion spectrum. With the ever-increasing accelerator power required to support high-energy physics initiatives, the pool of materials forming production targets or other critical components capable to withstand the pulse intensities associated with these multi-MW class machines is depleted. MW-class accelerator targets, in addition to the high proton fluence they must endure during their operational life, fluence that may approach those of reactor-based materials typically exposed to high neutron fluxes, must be able to continuously absorb and diffuse the severe thermo-mechanical shock resulting from the intense proton pulses. Tightly focused, high-energy proton pulses must be intercepted by the envisioned accelerator targets within several nanoseconds leading to extremely high deposited energy densities that are far beyond what most materials can tolerate. Densities of energy deposited under these conditions on the target as well as other beam-intercepting elements like collimators are estimated to be extremely high and far beyond what common materials, even those with extensive reactor-based track record, can tolerate. Further, the thermo-mechanical fatigue issue, direct result of the pulsed nature of these MW-class accelerators, adds additional performance requirements on these materials because of the need to extend the operational life of these components. Fatigue limits, even under the best of circumstances in which materials can resist irradiation-induced degradation of their properties, can be reached in short time stemming from the fact that accelerator pulse frequencies are of the order of 60 Hz. Therefore, utilization of beryllium as a pion production low-Z target in high power proton accelerators has been sought for two reasons namely (a) its low Z which desirable to produce a certain pion spectrum and (b) its good thermal properties that can be relied upon to mitigate the intense thermo-mechanical shock expected to be induced under the multi-MW power demand and the proton pulse structure.

Whether in a high energy neutron or energetic proton environments impinging protons and/or neutrons will knock out atoms in the beryllium crystal lattice generating defects, such as vacancies. Coalescence of vacancies leads to void formation and swelling adding to the swelling from the gaseous nuclear reaction products of He and H that are formed. A number of studies [3-6] have assessed that transmutation gas production plays a more important role than radiation-induced damage in the form of interstitial and vacancy formation in limiting the lifetime of the material. When irradiated at elevated temperatures, neutron or energetic proton irradiation is expected to cause He bubbles to form and embrittle grain boundaries. Studies [2] have also observed that beryllium irradiated at low temperatures (50°C) but subsequently annealed to 750°C exhibited significant swelling (~27%) attributed to large bubble formation. Embrittlement and helium swelling during low temperature irradiation according to studies [3-6, 14] should be limited for beryllium.

Beryllium being a hexagonal close-packed structure has only two operating slip planes (0001) & (1010) significantly limiting the ductility of the metal [15]. For this reason important engineering properties such as fracture toughness, ultimate strength, and plastic elongation has been thought to be poor. It appears however that some of these properties, particularly ductility, are functions of

fabrication process and controllability of grain boundary integrity which is associated with the removal of oxygen.

Several studies focusing on the properties of beryllium over a range of temperatures including irradiation effects from neutrons and in some cases energetic protons have been conducted in recent years. An excellent summary of unirradiated beryllium properties are presented by K. A. Walsh [16] where the physical, elastic and crystallographic properties as functions of temperature are described. The microstructure of out-of-pile annealed neutron irradiated beryllium was studied in [17] by X-ray tomography. Specifically, beryllium reflector fragments irradiated for 15 years in a research reactor at low temperatures were post-irradiation annealed and porosity was deduced after annealing using synchrotron X-ray micro-tomography. Studies of irradiated BeO have shown the recovery of the lattice parameters c and a [18]

This paper focusses on the effects of energetic protons and the damage they induce on Beryllium. Irradiation of beryllium samples that was one of the materials comprising a large array of candidate materials under study was performed at the Brookhaven National Laboratory Linac using 200 MeV protons. Following an exposure to peak proton fluence of $\sim 1.2 \times 10^{20}$ p/cm² the beryllium samples underwent post-irradiation evaluation focusing on the effects of irradiation damage on stress-strain characteristics, ductility and thermal expansion coefficient. In addition, and through high temperature annealing, the damage reversal as well as oxidation and transmutation gas mobilization and diffusion on volumetric changes were studied. X-ray diffraction studies on the effects of irradiation and macroscopic stress on the microstructural evolution and crystallographic role in the deformation of the beryllium were also conducted as part of the experimental campaign. This paper, however, presents only the effects of irradiation damage on the properties of beryllium observed macroscopically. The X-ray diffraction analysis findings of the damage induced by the 200 MeV protons on beryllium microstructure are presented in [19].

The experimental details and configuration of the beryllium sample array in the irradiating proton beam accompanied by estimated energy deposition and damage in the form of displacements-per-atom (DPA) are presented in the subsequent section. Estimates of energy deposition in the beryllium array are used to estimate through a 3-D thermo-mechanical analysis of the experiment in an effort to establish a good basis for comparison with studies by other researchers. The experimental details section is followed by the summary on the stress-strain behavior of the unirradiated Beryllium, the effects of the irradiation as a function of proton fluence and the role of the *hcp* crystal structure of Beryllium in the macroscopic elasto-plastic behavior. Subsequently, the effects of proton irradiation on the dimensional stability and the thermal expansion coefficient are presented and discussed along with the findings on volumetric changes and damage reversal via high temperature annealing. In the summary section the observations on proton irradiation effects on beryllium are summarized and discussed within the context of available data from similar or relevant studies.

2. Experimental

2.1 Microstructural Characterization and Sample Preparation

Brush-Wellman standard grade of beryllium S-200-F produced through the consolidation of beryllium powder via vacuum hot pressing was obtained and used in the experiment in the form of ready samples that were specifically designed for the experiment. The fabrication process utilized and measurement or specification of the composition of the actual material was unavailable upon receipt of the produced samples. In addition, prohibitive regulations in effect at the home institution where the experiments were conducted associated the handling of beryllium, prevented microstructural and metallurgical analysis upon receipt of the material specimens. Microstructural studies, however, were performed on the received S200-F beryllium grade utilizing both scanning electron microscopy and EDS on unirradiated/unpolished samples and X-ray diffraction on irradiated and as received S200-F beryllium. Figures 1(a; b; c and d) depict SEM micrographs of the unpolished surface of as-received beryllium obtained using the JOEL 7600F analytical high resolution electron microscope with the integrated INCA X-ray microanalysis system (EDX) at the Center of Functional Nanomaterials (CFN) revealing grain size, porosity and the presence of impurities at grain boundaries. Energy Dispersive Spectroscopy (EDS) analysis results are shown in Figure 2 (a-d). The spectral analysis revealed that oxygen is the dominant impurity in the form of BeO, followed by carbon, aluminum and iron as well as traces of Ni, Ca and Cu.

Figure 1: Scanning electron micrographs of the unpolished surface of S200-F beryllium shown a grain sizes with wide scattering ($<10\mu$), porosity and impurities over the grain boundaries.

Figure 2: Identification of impurities in the as-received S200-F beryllium using Energy Dispersive Spectroscopy (EDS) analysis. (a) Phase map and (b) EDS spectrum

To further reduce uncertainties regarding the microstructure of the received S-200-F grade beryllium, experimental data from X-ray diffraction analyses performed at the NSLS synchrotron on both unirradiated and proton-irradiated beryllium following the irradiation phase were used. While the X-ray diffraction studies were conducted to understand beryllium lattice changes as result of irradiation and loading that leads to residual strain and activates twinning they also aided in (a) the identification of impurities such as BeO present at levels where crystallographic reflections can be observed and (b) in the estimation “average” grain sizes and crystal sizes. The two-fold X-ray diffraction experiments consisted of (a) 70 keV monochromatic X-rays and XRD technique and (b) polychromatic 200 keV X-rays and energy dispersive diffraction (EDXRD) technique and were conducted at the National Synchrotron Light Source (NSLS). Confirmed by the two X-ray diffraction studies is that BeO is the

dominant impurity in the S-200-F beryllium received experiencing no changes after irradiation. Analysis of the EDXRD diffraction peak intensity variation for several of the beryllium crystallographic planes obtained by scanning over the 1mm sample thickness for $2\theta=4^\circ$. The gauge volume is defined by the interception of the incident beam and the diffracted beam path. For the current experiment characterized by $30\mu\text{m} \times 1000\mu\text{m}$ beam and 1.5mm depth into the sample the gauge volume is $4.5 \times 10^7 \mu\text{m}^3$ leading to an estimate of minimum grain size of $\sim 4.2\mu\text{m}$ and confirming that (a) the S200-F beryllium received and studied under proton irradiation is a “fine-grain” beryllium grade and (b) that the grain size has a wide scattering as also shown in the SEM micrographs. Excerpt X-ray diffraction data on the S200-F beryllium used for microstructural characterization are presented in a later section to compliment the macroscopic study objective of the irradiated beryllium. A detailed study of the irradiation-induced changes in the S200-F beryllium microstructure based on X-ray diffraction and the use of high energy monochromatic and white X-rays as well as microstructural analysis based on EDS/WDS and Electron Backscatter Diffraction (EBSD) is included in [19].

Two types of sample designs were utilized in the experiments from the vacuum hot pressing material. One type, termed tensile, was to be used in stress-strain and eventually X-ray diffraction analysis under a four-point bending state of stress. The second type, termed CTE, was to be used in thermal analysis and high temperature annealing. The specific design of the two types of samples was dictated by the need to eliminate any shining paths in the direction of the proton beam. This resulted in a high precision manufacturing process of the samples. The geometric and shape details of the two samples are shown in Figure 3. The 1mm thick tensile sample is of a special “dog-bone” shape with a length of 29.5 mm between the grips. The effective “gauge” length is 6 mm. The CTE-type sample is 3mm thick, has a total length of 29 mm and a central section whose contour exactly matching and mating with the gauge contour of the tensile-type sample.

Figure 3: Shape and dimensional details of the two types of specimens utilized in the proton irradiation of Beryllium study (a) tensile, and (b) CTE specimens.

Typical property values for the S-200-F beryllium grade are reported as: yield strength ≥ 241 MPa; ultimate tensile strength ≥ 324 MPa; elongation at break $> 2.0\%$ and Poisson's ratio $\nu = 0.18$. The smaller, however, is the grain size, the greater the ductility and the strength of beryllium. Company data for the finer grain ($\sim 6 \mu\text{m}$) are reported having yield strength of 329 MPa, ultimate tensile strength of 492 MPa and tensile elongation 7.3%.

In an effort to deduce baseline properties prior to irradiation of the beryllium received, given the variability and range in exhibited mechanical and especially tensile elongation values, mechanical tests were performed in room temperature using a Tenius-Olsen, 5000N mechanical tester situated within a hot cell facility and under the exact same conditions the irradiated samples were to be tested. Figures 4 (a & b) depict the results of the stress-strain tests which indicate the grouping of the as-received specimens into two distinct sets, a variation that attributed by the authors on grain size variation between material lots that the samples were produced from. Within each set a excellent reproducibility

of the stress-strain behavior is observed indicating that the material fabrication has achieved uniformity. The tensile elongation, yield strength and ultimate strength observed in these mechanical tests confirm the results of the X-ray diffraction analyses that the material is indeed fine-grained.

Figure 4: Stress-strain behavior of unirradiated S-200 F grade Beryllium. Arrows indicate locations of possible twinning activation in the hcp material. The difference observed in the stress-strain behavior of the two batches is attributed to the variation of grain size between material lots.

Kinks occurring at the same stress levels for both sample groups are observed may be attributed to the *hcp* lattice structure of beryllium and the deformation or slip modes that are associated with them. At room temperature the basal slip or twinning influences the deformation and the level of ductility it can

be achieved. The twinning deformation mode is associated with the extension in the c lattice direction requiring high shear stresses for its activation and representing the dominant mechanism behind inelastic shape changes along the c -direction which macroscopically may appear as plastic deformations in the stress-strain curve. While the deformation originates in the beryllium lattice it also macroscopically observable as an integrated effect for a large number of crystals. What one observes with these stress tests is the occurrence of “kinks” which tend to enhance ductility at specific stress levels [20, 21]. Further, the data show that the “kinks” appear to occur at same stress levels for both material groups suggesting that texture variation is not a contributing factor in the different mechanical properties.

2.2 Proton Irradiation Experiment

A set of beryllium specimens of the types shown in Figure 3 was assembled in a special capsule and inserted, along with a number of other test materials, in the path of the 200 MeV protons from the BNL Linac. The irradiation was conducted under $\sim 10\text{m}$ of water. The beryllium assembly is depicted in Figure 5. The precise design and fabrication of the specimens allowed for the closely meshed array. Three (3) 1mm tensile samples were placed in each tensile position and one 3mm-thick CTE sample for its corresponding position. Twenty one (21) tensile beryllium samples were used in the irradiation array along with six (6) CTE samples. The beryllium samples were cooled by flowing water past the front face of the first layer samples. The proton beam intercepted by the array is in the form of a 2-D Gaussian and therefore there exist (a) variation in the fluence received per sample and (b) consequently variation in the irradiation temperature of each tensile and CTE sample of the array.

The total number of protons incident on the beryllium sample array was $\sim 5.27 \times 10^{20}$ (integrated beam current $\sim 23,453 \mu\text{A}$ -hours). The proton energy in the beryllium array ranged between 168 and 162-MeV. The proton beam exhibited a Gaussian transverse profile with $\sigma_x = \sigma_y = 9.4 \text{ mm}$ and had an x-y offset from the center of the array. Special nickel foils integrated into the irradiation assemblies were analyzed and helped establish the beam spot size, shape and position during irradiation. Because monitoring of the irradiation temperature during beam exposure was not feasible due to the hermetically sealed configuration for the sake of medical isotope production downstream of the test material array, the information on the shape and position of the beam is vital because the study relied on numerical analyses to deduce irradiation temperatures and the fluence profile for the array of samples. Using the transport codes FLUKA [22, 23] and MCNPX [24] the interaction of protons with the test material matrix array that included the beryllium capsule of Figure 5 were conducted leading to (a) energy deposition distribution per sample and (b) irradiation damage expressed in Displacements-per-Atom (DPA) per sample. Details of the 3-D numerical model used in FLUKA that reproduces the beryllium assembly during irradiation is shown in Figure 6. The beryllium samples in addition to the protons are exposed to a neutron flux from the spallation process in the upstream test materials of the irradiation array such as Vascomax and Ti-6Al-4V. The spallation neutron flux however is approximately two orders of magnitude lower than the flux of the primary protons and therefore the irradiation damage stems primarily from protons. The energy deposited (electronic interaction) in the Beryllium array was calculated to be of the order of 248 W/cm^3 (peak). Based on the beam profile and position during irradiation it was deduced that the peak fluence in the array was experienced by one of the CTE type specimens ($\sim 1.2 \times 10^{20} \text{ p/cm}^2$). The peak fluence in the “dog-bone” tensile specimens was estimated to be $\sim 1.09 \times 10^{20} \text{ p/cm}^2$. The peak damage in DPA achieved during the 200-MeV proton irradiation was estimated by the transport Monte Carlo codes to be $\sim 0.35 \text{ DPA}$.

Thermal analyses were performed based on 3D models reflecting the exact geometric configuration while accounting for contact conditions between the samples. Additionally, computational fluid dynamics (CFD) analyses were performed to estimate the convection film coefficients in the cooling channels between the various material layers. Proton beam energy deposition on the beryllium array was deduced from the FLUKA transport code [22, 23] and used for the estimation of irradiation temperatures throughout the array. The estimated temperatures varied between 100°C - 180°C

depending on the position of the sample relative to the proton beam. The irradiation of beryllium achieved in this experiment may be classified as low temperature irradiation for the purpose of direct comparison with other studies.

Figure 5: Be sample array placed in the path of the 200 MeV irradiating proton beam. Beam energy at beryllium array ranges between 168-162 MeV. One of three tensile specimen layers and their respective position/ numbering is shown in (a) as B-1, B-2, etc. In (b) the complete array consisting of three tensile samples per position integrated with 3mm-thick CTE samples are shown.

Figure 6: Three dimensional (3D) numerical models precisely mimicking the beryllium array experimental configuration (Figure 5) used by the transport code FLUKA. Peak fluences estimated to be 1.2×10^{20} p/cm² and 1.09×10^{20} p/cm² in the CTE and tensile samples respectively.

2.3 Post-irradiation analysis

The post irradiation evaluation (PIE) of the Beryllium array was multi-faceted and it included macroscopic and microscopic evaluation of the effects of proton irradiation on key properties of the material. The macroscopic assessment, which is the subject and presented in this paper, included stress-strain behavior, irradiation induced ductility loss, dimensional stability, thermal expansion coefficient variation and damage reversal through high temperature annealing. Microstructural effects of irradiation and stress, including Beryllium crystal deformation, post-fracture lattice state and inherent anisotropy were studied using monochromatic and polychromatic high energy synchrotron X-ray beams using XRD and EDXRD techniques. Only excerpt results of the microscopic studies [19] are reported in this paper and used as basis in explaining macroscopic observations on the deformation of irradiated and unirradiated Beryllium that are stemming from its *hcp* lattice crystal structure.

The macroscopic phase of the PIE was performed at the BNL hot cell laboratory facility. Prior to the thermal analysis and mechanical testing of the Beryllium specimens their activation was measured so samples and position to the beam (fluence) can be verified. Additionally, γ -spectroscopy was performed on representative specimens for isotopic composition. The inserted nickel films positioned within the irradiation matrix were autoradiographed to establish the beam spot size, shape and position relative to the specimens. The nickel film characterization revealed that the beam was off-centered as schematically shown in Figure 6.

2.3.1 Mechanical Testing

The tensile stress-strain tests, similarly with the unirradiated dog-bone samples reported in Section 2.1 that helped establish the baseline and the properties of the as-received material were performed remotely in room temperature inside the hot cell. Similarly, the Tenius-Olsen 5-kN maximum load tester was used at the same strain rate of 1mm/minute. Stress-strain test results of irradiated Beryllium tensile samples are shown in Figures 7 and 8. In Figure 7a the tensile samples depicted on Figure 5 (B1-B7) selected irradiated samples were compared directly with the unirradiated case (numbering corresponds to each marked Be sample that was irradiated with a sample-specific proton fluence over its 6mm gauge based on its spatial position relative to the proton beam position). Fluence associated with each specimen has been deduced and it represents the peak fluence experienced over the gauge of each tensile specimen. Specifically, and based on the fact that there was an actual beam offset, the peak fluence in tensile specimens was estimated to be $\sim 1.09 \times 10^{20} \text{ p/cm}^2$ and was experienced by samples in the center position of the array (i.e. B-4 in Fig. 5). It should be noted that the irradiation temperature varies from specimen to specimen as a function of its position relative to the beam and range between 180°C at the proton beam center to $\sim 100^\circ\text{C}$ at the beam 3σ .

The tensile test results of all the specimens forming the three layers in the array (7 per layer, 21 tensile specimens in total) and covering the entire range of received fluence, or irradiation damage, reveal the following: As expected from experience stemming from typical metals, irradiation induced changes in the mechanical behavior of beryllium as a function of damage or fluence. Ductility loss accompanied by yield and ultimate tensile strength increase is observed. For the S200-F beryllium, however, the yield stress and subsequently the post yield or plastic zone is not clearly delineated as in other metals (shown in Figure 7). Further, it is observed that scattering exists on the elongation at break data stemming possibly from either the variation in the microstructure or the cut orientation in the specimens as discussed in Section 2.1. For the irradiation or fluence level of $\sim 1.09 \times 10^{20} \text{ p/cm}^2$ the loss of elongation at break is of the $\sim 30\%$. The beryllium in the irradiated state still maintains similar post-yield behavior where the strength continues to increase to the ultimate tensile strength. Noted in Figure 7b is the presence of stress kinks that were attributed to the crystal twinning deformation mode and they are observed in both unirradiated and irradiated samples. The observed kinks appear to be activated in the irradiated materials at higher stresses and monotonically with irradiation fluence.

Discussion on the correlation of the activation of twinning deformations in the beryllium lattice with these macroscopically observed features during tensile stress tests of the hcp structure is included in the summary section of this paper.

Figure 7: Tensile stress-strain test results of proton irradiated Beryllium

Figure 8: Fractional change of Young's modulus of proton irradiated beryllium with proton fluence.

Results of tensile tests on all twenty one (21) irradiated S200-F beryllium specimens (7 per layer) were used to deduce changes in Young's modulus as a function of received proton fluence. In the ensemble, three samples were exposed to the same fluence. Figure 7a depicts one layer of samples with the corresponding fluence per sample. It should be pointed out that wide scattering was observed in the

stress-strain data generated stemming from the different material lot the samples were made from and the variation in grain size and possibly the slight variation in irradiation temperature that existed between samples. The influence on the increase in the Young's modulus as a function of fluence is depicted in Figure 8.

2.3.2 Annealing, Thermal Stability and CTE

Using the horizontal, two-rod LINSEIS dilatometer, a comprehensive thermal analysis was performed that consisted with thermal cycling and high temperature annealing. The objective of the thermal cycling was to assess dimensional changes that will in turn explain, when compared with the tests on the as-received material, the effect of temperature in (a) the stabilization of the microstructure which is expected to exhibit some degree of porosity (b) the thermal strain behavior of beryllium over different temperature ranges, (c) the variation of linear thermal expansion coefficient and finally (d) the thermal activation time or number of thermal cycles required for annealing of irradiation-induced damage. Thus, prior to high temperature thermal annealing phase which included a number of isotherms in both "heating" and "cooling" stages, two-phase thermal cycling was performed on the irradiated CTE-type Beryllium specimens and compared to the unirradiated state. During the first phase up to four (4) thermal cycles with peak temperature of 300°C were conducted followed by up to four (4) cycles with peak temperature of 600°C. No dwell time was introduced into these thermal cycles.

Figure 9 depicts successive thermal cycling of unirradiated Beryllium to 610°C peak temperature and Figure 10 shows successive thermal cycling of proton-irradiated beryllium to 310°C and 610°C. Based on the dimensional changes recorded for the 29mm long samples an "apparent" non-linear thermal expansion behavior in the low temperature regime (<160°C) is observed and only during the heating part of the thermal cycle. The thermal cycle is characterized by 2°C/minute rise during the heating phase and 5°C/minute during the cooling phase of the cycle. Following experimental efforts specifically conducted for assessing the potential contribution of the apparatus response to the apparent "knee" by tracing the identical thermal cycle using a quartz sample with known, extremely low thermal expansion, it was assessed that the "knee" is most-likely the result of temperature lag in the sample during heating. This assertion is further supported by the fact that the heating and cooling rates in these experiments were relatively high (2 and 5°C/minute respectively). While "non-linear" thermal expansion trends in Beryllium and in particular within this temperature range have been reported [16] both for macroscopic behavior and lattice thermal expansion, the degree of "non-linearity" observed in these experiments is such that it points to thermal lag in the test sample during heating as a significant contributing factor to the "knee" shown.

Figure 11 (a,b,c) depict thermal cycling results of irradiated beryllium samples exposed to 0.2×10^{20} , 0.3×10^{20} and 0.05×10^{20} average fluence respectively. Average fluence values were deduced based on the beam profile over the entire sample volume for each CTE sample and used in representing the experimental data. Thus, while the peak fluence identified to occur over a sample is $1.2 \times 10^{20} \text{ p/cm}^2$, the corresponding average fluence characterizing the CTE sample is $\sim 0.6 \times 10^{20} \text{ p/cm}^2$. This was done to account for the fluence variation along the length (not uniform) of the CTE sample given that the dimensional changes measured by the dilatometer are the integrated effect along the length.

As shown in Figure 11, the irradiation damage manifested as variation in thermal expansion is mostly reversed for the temperature regime (20-310°C) even after a single thermal cycle to 310°C. This implies that interstitial atoms displaced from their position in the basal planes of the lattice receive enough activation energy provided by the annealing temperature (310°C in this case) to return to the lattice basal planes. Interstitial atoms displaced by the irradiating protons further from the basal plane remain as interstitials until they are activated by higher annealing temperature. The process is accompanied by the shrinking along the *c* axis inducing volumetric change. Subsequent thermal cycling to 610°C results in more interstitials getting mobilized and returning to sites on the basal plane. Figure 11(c) shows thermal cycling to the higher 610°C temperature by comparing irradiated and

unirradiated beryllium samples. From the “relative” dimensional or volumetric changes measured it is evident that there is no 100% recovery after annealing to 610°C (damage recovery measured as thermal expansion similarity to that of the unirradiated state shown in Figure 11c). This confirms similar findings obtained during the study of BeO [26] after neutron irradiation regarding the mobilization of the recovery of lattice *a* and *c* parameters with increasing temperature. Figure 12(a,b) depict the effects of irradiation on thermal expansion and annealing through cycling at higher dose (average fluence $\sim 0.6 \times 10^{20}$ p/cm²). Clearly shown during the first 610°C thermal cycle that there is a significant relative length (and thus volumetric) reduction which is explained as recovery of interstitials which get thermally activated and mobile and return to the basal planes of the beryllium lattice inducing shrinkage of the *c* and lattice parameters. As its observed, subsequent thermal cycles to the same 610°C temperature result in no further volumetric change given that all the interstitials that could have been activated have returned to the basal plane. As noted in [2] the process is fast and it does not require long dwelling at an activation energy level or temperature to be completed.

Depicted in Figures 13 and 14 are results of average and instantaneous thermal expansion coefficient (CTE) generated from the cyclic thermal annealing of unirradiated and proton irradiated beryllium. Average CTE for the unirradiated S200-F following “stabilization” via annealing, i.e. TC3 cycle for temperatures <100°C is of the order of $\sim 11 \times 10^{-6}$ K⁻¹. Similar values for the low temperature regime (20-65°C) were reported by Walsh [16] for unirradiated S200-F. Also reported in [16] is the non-linear increase of the average CTE to $\sim 18 \times 10^{-6}$ K⁻¹ for the range of 25-1000°C which is also confirmed by this study. The non-linear behavior of the thermal expansion and thus of CTE at the lower temperature range (<200°C) that is exhibited by the S200-F studied in this paper was further scrutinized to identify potential system/apparatus effects. Specifically, studies using the identical thermal profile in the LINSEIS dilatometer and a quartz calibrating rod with extremely low and known thermal expansion coefficient indicated that the particular knee that appears in the thermal expansion of beryllium must have its origin in the material itself and is attributed by the authors to be the result of lattice anisotropy in the hcp material structure. The variation of the CTE as a function of proton fluence is depicted in Figure 15.

Figure 9: Thermal cycling to 610°C of unirradiated beryllium

Figure 10: Thermal cycling to 310°C and 610°C of proton-irradiated beryllium (average fluence over sample: $0.43 \times 10^{20} \text{ p/cm}^2$)

Figure 11: Dimensional change and annealing observed over thermal cycling to 310 $^{\circ}\text{C}$ (a & b) followed by (c) thermal cycle to 610 $^{\circ}\text{C}$ of irradiated beryllium for irradiated beryllium samples to different average fluences.

Figure 12: Comparison of the relative dimensional change and annealing through thermal cycling in irradiated beryllium (average fluence over test sample: $0.6 \times 10^{20} \text{ p/cm}^2$)

Figure 13: Average Thermal Expansion Coefficient (CTE) of irradiated and unirradiated beryllium.

Figure 14: Evolution of thermal expansion coefficient (instantaneous) of irradiated beryllium with thermal cycling compared with as received material. TC_i (i=1-3) indicates thermal cycles. Results shown are for the heating phase of the cycle

Figure 15: Instantaneous Thermal Expansion Coefficient (CTE) of irradiated and unirradiated Beryllium at 550°C as a function of average (averaged over the 29 mm long CTE type sample) proton fluence. The error shown indicates variation observed from repeated measurements stemming from thermally induced changes in the microstructure with thermal cycling.

2.3.3 High temperature annealing

Subsequent to the thermal cycling and CTE evaluation phase high temperature annealing was conducted in air to temperatures up to 740°C. While irradiation took place at low temperatures (<200°C) the high temperature annealing aimed to mimicking higher irradiation temperature response by observing macroscopic dimensional changes in the irradiated beryllium. A detailed assessment of the competing mechanisms in the volumetric changes and of the role of irradiation can be found in [2].

Shown in Figure 16(a) is a multi-isotherm annealing of unirradiated beryllium. The experiment involved 6-hour dwell times for the different temperature levels up to 730°C and for 10-hour dwell time over the 740°C isotherm. The objective was to qualify and quantify the volumetric change that occurs as a result of manufacturing defects in the microstructure and porosity. It is apparent that at temperatures $\geq 600^\circ\text{C}$ beryllium undergoes contraction during the heating part of the annealing cycle. In the cooling part of the annealing cycle ($\leq 700^\circ\text{C}$) the beryllium volume remains stable indicating the microstructural defects have been annealed due the higher level temperatures. Figure 16(b) and (c) demonstrate dimensional changes occurring during heating and cooling over the same temperatures.

Figure 16: Long thermal annealing of unirradiated beryllium with 6-hr and 10-hr isotherms to 740°C

To make a direct comparison with the irradiated beryllium shown Figure 17 depicts dimensional changes over the 500°C isotherm subsequent to the thermal cycling discussed in the previous section to temperatures up to 600°C. As anticipated from the damage annealing that was triggered through thermal cycling the irradiated samples exhibit dimensional stability at temperature isotherms <600°C.

Figure 17: Dimensional stability over a 500°C isotherm during annealing and following the completion of thermal cycling to 300°C and 600°C.

The dimensional change at higher temperatures (>600°C) where a number of competing mechanisms are at play, i.e. damage annealing through mobilization of interstitials accompanied by *c* and *a* lattice parameter changes, transmutation gas mobilization and diffusion resulting in swelling, and oxidation, are depicted in Figures 18 and 19. In Figure 18 the dimensional changes of an unirradiated (pristine) beryllium sample, an unirradiated sample that underwent thermal cycling to 600°C and an irradiated sample with fluence of 0.2×10^{20} p/cm² are directly compared. What is observed is that, following the initial annealing during the heating part of the cycle of the microstructural imperfections, the unirradiated beryllium samples exhibit identical slopes. The offset is simply the thermally activated dimensional adjustment from the prior thermal cycling.

Irradiated beryllium, on the other hand, which also underwent thermal cycling similar to the unirradiated sample experiences volumetric changes at different rate than its unirradiated counterpart. The first steep part of the 700°C and 730°C isotherms is attributed to damage annealing accompanied by porosity evolution. The flattening and eventual upswinging of the trace appearing at $T > 730^\circ\text{C}$ and in particular 740°C isotherm (as clearly seen in Figure 18b) is attributed by the authors to He and H mobilization/diffusion and coalescence into bubbles which in turn induce swelling. Large amount He and H are expected to be produced by the 160 MeV protons that irradiated the S200-F beryllium under study. It is estimated that at these proton energies ~4000 appm/DPA of He gas is produced. This corresponds to ~470 appm He for the irradiated sample in Figure 18. It is deduced from Figure 18b that the triggered swelling overtakes the contraction caused by the other mechanisms (defect annealing, oxidation and porosity reduction). This is supported by the fact that over the same annealing isotherm of 740°C (shown in Figure 19) and for higher damaged beryllium where one would have expected defect annealing rate to induce flattening at temperatures 10°C lower (two isothermal annealing stages at 730°C denoted as C¹ and C² in Figure 19) no flattening is observed indicating continuous annealing of irradiation induced defects. Flattening for the higher irradiation sample of beryllium only begins to occur during annealing at 740°C (see stage D in Figure 19). A subjective

explanation of the governing processes stemming from the annealing behavior at these high temperatures is that when annealing is applied to irradiated beryllium exposed to higher proton fluence (i.e. CTE sample with average fluence of $0.6 \times 10^{20} \text{ p/cm}^2$), the competing mechanisms governing the dimensional change at temperatures $>600^\circ\text{C}$ are in different order.

Figure 18: Effects of thermal annealing on unirradiated and proton irradiated beryllium ($0.2 \times 10^{20} \text{ p/cm}^2$) at elevated temperatures. Depicted is the volumetric change reversal from contraction to expansion over the 740°C isotherm for the irradiated Be at the shown dose.

Two (2) back-to-back thermal annealing cycles were performed each consisting of several isotherms both during the heating and the cooling parts of the thermal cycle. The first cycle reached the temperature of 730°C and the second 740°C. The important things to note are the following: Over the high temperature isotherms the restoration of the lattice through annealing of the damage (interstitial and defect mobilization) resulting in volumetric contraction dominates even upon repeat of the thermal cycle. This indicates that the duration of the annealing over these high temperature isotherms is important and it dominates the process. Only over the 2nd thermal annealing cycle and at the highest isotherm (740°C) the swelling from the transmutation gas diffusion is evident. Based on the observations of Figures 18 and 19 it is assessed that the temperature region between 730 and 740°C is of significance in the microstructural evolution of irradiated beryllium, a finding that is in agreement with previous studies [2] on out-of-pile neutron irradiated beryllium where mobilization of transmutation gasses accelerates over a narrow temperature interval. In this study it is assumed, based on dilatometric observations that the threshold temperature is ~740°C.

Figure 19: Effects of thermal annealing on proton irradiated beryllium with average fluence over the test CTE sample of $0.6 \times 10^{20} \text{ p/cm}^2$ at elevated temperatures and over two consecutive thermal cycles TC1 and TC2 consisting of multiple isotherms during heating and cooling phases. Notations A^i , B^i , C^i , D ($i=1, 2$) shown indicate annealing over isotherms 600°C, 700°C, 730°C and 740°C respectively.

3. Summary and Discussion

A 200 MeV proton irradiation experiment was performed on S-200F grade beryllium specimens specially designed for post-irradiation mechanical and thermal testing to the peak proton fluence of $1.2 \times 10^{20} \text{ p/cm}^2$. Owing to the proton beam profile during irradiation and the layout of the beryllium samples, a variation of fluence was achieved that enabled the assessment of fluence vs. irradiation damage in post-irradiation. For thermal stability and post-irradiation analysis average fluence levels were deduced for each test CTE-type specimen. Peak fluence estimates used to characterize tensile-type samples were deduced from the proton beam profile over the gauge of the sample. Irradiation temperatures were considered low and were in the range of $100^\circ\text{C} - 180^\circ\text{C}$.

3.1 Irradiation effects on S200-F beryllium microstructure

In an effort to “connect” macroscopic observations made on proton irradiated beryllium (macroscopic assessment being the objective of the present study), and in particular regarding the characteristic stress-strain behavior of the hcp beryllium which exhibited a series of “kinks” during the tensile tests where the material appeared to undergo local yielding at specific stress levels, X-ray diffraction data were used from post-irradiation EDXRD and XRD studies that were conducted. These studies provided a qualitative assessment of the impurities present which was cross-correlated with the microstructural analysis using SEM/EDS, grain size estimates which were also correlated with microstructural characterization, assessment of potential preferred orientation, and most importantly provided a microscopic view of the characteristic stress-strain behavior observed during the tensile stress tests. Only excerpt results of the multi-faceted X-ray diffraction experiment are presented here for the sake of correlation with macroscopic observations. Figure 21 depicts a 3-D EDXRD phase map of an irradiated tensile-type beryllium specimen exposed to a peak fluence of $0.97 \times 10^{20} \text{ protons/cm}^2$ in post-fractured state (i.e. following tension test to failure).

Figure 20: EDXRD map of proton irradiated beryllium in post-fracture state and scanning across the fracture zone of the tensile specimen. Shown are both the reflections from BeO present in the material matrix, the scatter of the crystallographic plane reflection intensities used in grain size estimation via EDXRD and the evolution of the crystallographic planes Be(002) and Be(102) under tension leading to eventual fracture.

Shown in Figure 20 is X-ray scanning across the tensile gage of the specimen (dog-bone neck shown in Figure 3a) where there exist a variation in irradiation damage stemming from fluence variation, the fracture zone where the X-ray beam with width of 1000 μ m and height of 30 μ m is able to scan across and the zone on either side of the fracture surface where plastic deformation has occurred. The triggering of different deformation mechanisms at the lattice/microstructural level that occur during the tension test are expected to be observed in the crystallographic reflections and therefore can be correlated with the macroscopically observed behavior. The particular relation between the basal plane reflections and those of twinning activation governed by the reduction of the intensity in Be (002) and accompanied with increase in the Be (102) reflections is expected to occur in this hcp structure. Evidence of the mechanism of disappearance of (002) reflection due to twinning activation occurring in hcp structures and in particular beryllium can also be found in [29].

Figure 21: X-ray diffraction analysis of unirradiated and irradiated beryllium confirming via a microscopic analysis (a) the presence of impurities that exist in the material and up to levels that distinct diffraction peaks can be identified and (b) the resilience to irradiation damage for fluences up to 1.09×10^{20} protons/cm² of the fine-grain S200-F beryllium as observed via the macroscopic analysis.

Figure 21 depicts X-ray diffraction data of unirradiated and proton irradiated beryllium with identified phases and the effect of proton fluence on the lattice structure. While impurities of quantity that can be detected by crystallographic reflections are identified in agreement with the microstructural analysis, it is important to note that S200-F beryllium experiences limited changes in its lattice structure, an observation that is in line with the macroscopic analysis.

In post-irradiation analysis, and following activation measurements to confirm in conjunction with the special foil introduced in the irradiation array the beam position relative to each array sample, a series of macroscopic and x-ray diffraction experiments were conducted to understand and quantify irradiation damage effects of energetic protons on beryllium. In this paper the macroscopic results of the study and in particular (a) the stress-strain behavior and ductility loss, (b) the thermal expansion and stability of irradiated beryllium were studied for the temperature range between 20°C and 740°C and the evolution of thermal expansion coefficient variation in the temperature range 60°C-740°C and (c) the annealing of irradiation damage via long duration, multi-isotherm thermal treatment are presented.

3.2 Irradiation effects on stress-strain behavior and ductility

- In its as-received, unirradiated state the S-200F beryllium exhibits up to 6% elongation to fracture which indicates that it is made of fine grain structure. The delineation of the elastic and the plastic deformation regimes it is only faintly observed. What is important to note is that beryllium, having an *hcp* crystal structure, exhibits deformation twinning that can be inferred by macroscopic

observations over the elastic and into the plastic regimes. Microscopic analyses based on scanning electron microscopy and augmented by electron backscatter diffraction and XRD analysis using 70 keV monochromatic x-rays showed no preferred grain orientation. Based on this microscopic observation it is assessed that the differences in stress-strain and ductility behavior of the groups of test samples are potentially linked to differences in grain size between different material lots the test samples were made from.

- In its irradiated state, tested at room temperature, and for the damage levels that have been reached beryllium appears to resist serious ductility loss due to the irradiating protons while at the same time it experiences some increase in strength and elasticity modulus. Deformation twinning, manifesting itself as kinks, seeing as plastic-like deformations occurring during tensile testing, is still observable and characterized by an offset to a higher stress values. Microscopic analysis of residual twinning deformation using high energy x-rays and energy dispersive x-ray diffraction techniques confirmed this deformation mechanism and will be discussed in more detail in a dedicated paper [19].

3.3 *Irradiation effects on Thermal Expansion Coefficient and Dimensional Stability*

- As observed in this experiment, thermal cycling indicates that at temperatures $<200^{\circ}\text{C}$ beryllium thermal expansion is characterized by a “non-linear” relationship that is more pronounced during the heating phase of the cycle. It is assessed that temperature lag in the test sample during the experiments resulting from high heat rates ($\sim 5^{\circ}\text{C}/\text{minute}$) may have contributed to the observed non-linear relationship with temperature.
- Irradiation damage by energetic protons induces anisotropic but limited volumetric changes (swelling) resulting from interstitial atoms occupying the space between the c planes in the beryllium lattice. Thermal cycling to temperatures up to 610°C restores a significant portion of the volumetric change by thermally activating interstitial atoms. Comparison with the behavior of unirradiated beryllium revealed that a single thermal cycle is sufficient for restoring most of the thermal expansion behavior of the irradiated beryllium for the temperature regime below 610°C .
- The thermal expansion coefficient (CTE) for both unirradiated and irradiated beryllium exhibits a positive slope during heating in the temperature range $200\text{--}610^{\circ}\text{C}$ and is steady during cooling for the same range.
- Assessment of the relationship between proton fluence and CTE showed that the coefficient increases only slightly with increased fluence indicating that the beryllium microstructure is resistant to irradiation damage.

3.4 *Thermal Cycle Annealing*

The multi-stage isothermal annealing experiments on unirradiated and irradiated beryllium revealed the following:

- Unirradiated beryllium undergoes a “densification” of its microstructure over isotherms $>500^{\circ}\text{C}$ indicating the removal of fabrication defects and voids which are present in the as-received material.
- For proton irradiated beryllium a number of competing mechanisms governing dimensional change are identified in addition those that are common with the unirradiated counterparts. These are damage annealing accompanied by volumetric contraction, trapped transmutation gas mobilization/diffusion at temperatures $>700^{\circ}\text{C}$ accompanied by swelling and lastly oxidation at temperatures $\geq 500^{\circ}\text{C}$ characterized by volumetric expansion. Dominant volumetric change process, however, is the one associated with the irradiation damage annealing or lattice restoration leading to volumetric contraction.

- Irradiation damage via isothermal annealing is shown in the study to be a fast process in complete agreement with the observations made during thermal cycling and the recovery exhibited during a single thermal cycle.

4. Conclusion

Low temperature (100-180°C) irradiation of S-200F beryllium grade with energetic protons to peak fluence up to 1.2×10^{20} p/cm² have shown that in general beryllium exhibits resistance to radiation damage. This is demonstrated by the level of damage annealing observed via thermal cycling and high temperature annealing.

The as-received material contains manufacturing defects and porosity which can be removed with thermal annealing at temperatures >500°C. Tensile testing of unirradiated, as-received beryllium shows that this beryllium grade does not suffer significantly from brittle behavior but it exhibits tensile elongations as high as 6% which, along with the tensile strength and ultimate strengths, confirms the manufacturer's assigned values for a fine grain beryllium. The tensile tests also hint to microstructural deformation mechanisms that occur in this polycrystalline beryllium as a result of its *hcp* crystal structure.

When irradiated with energetic (~160 MeV) protons the S-200F beryllium loses only some of its exhibited ductility. High energy proton irradiation of beryllium leads to formation of point defects in the form of interstitials and vacancies as well as gaseous products of nuclear reactions as ⁴He and ³H. The irradiation damage in the form of interstitials and vacancies is annealed with temperatures above the irradiation temperature inducing volumetric contraction but the gaseous products begin diffusion and coalescence at elevated temperatures (≥740°C) leading to swelling. Irradiations with proton fluences $\sim 1.2 \times 10^{20}$ cm⁻² affect only slightly the thermal expansion coefficient (CTE) of beryllium.

Microstructural analysis post irradiation relying on high energy x-rays (both monochromatic and polychromatic) and techniques such as EDXRD and XRD revealed the limited damage induced by energetic protons on the S200-F microstructure. These findings are in agreement with the macroscopically observed post-irradiation behavior in terms of dimensional stability and stress-strain behavior.

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