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Failure Analysis of Beryllium Tile Assembles Following High Heat Flux Testing for the ITER Program

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Abstract

The following document describes the processing, testing and post-test analysis of two Be-Cu assemblies that have successfully met the heat load requirements for the first wall and dome sections for the ITER (International Thermonuclear Experimental Reactor) fusion reactor.

Several different joint assemblies were evaluated in support of a manufacturing technology investigation aimed at diffusion bonding or brazing a beryllium armor tile to a copper alloy heat sink for fusion reactor applications. Judicious selection of materials and coatings for these assemblies was essential to eliminate or minimize interactions with the highly reactive beryllium armor material. A thin titanium layer was used as a diffusion barrier to isolate the copper heat sink from the beryllium armor. To reduce residual stresses produced by differences in the expansion coefficients between the beryllium and copper, a compliant layer of aluminum or aluminum-beryllium (AlBeMet-150) was used. Aluminum was chosen because it does not chemically react with, and exhibits limited solubility in, beryllium. Two bonding processes were used to produce the assemblies. The primary process was a diffusion bonding technique. In this case, undesirable metallurgical reactions were minimized by keeping the materials in a solid state throughout the fabrication cycle. The other process employed an aluminum-silicon layer as a brazing filler material. In both cases, a hot isostatic press (HIP) furnace was used in conjunction with vacuum-canned assemblies in order to minimize oxidation and provide sufficient pressure on the assemblies for full metal-to-metal contact and subsequent bonding.

The two final assemblies were subjected to a suite of tests including: tensile tests and electron and optical metallography. Finally, high heat flux testing was conducted at the electron beam testing system (EBTS) at Sandia National Laboratories, New Mexico. Here, test mockups were fabricated and subjected to normal heat loads to 10 MW/m^2 (3 Hz) and abnormal heat loads to 250 MJ/m^2 (0.5s) to determine their performance under simulated fusion reactor conditions for first wall components. Both assemblies survived the normal heat loads with no visual damage. Optical and electron microscopy were used to evaluate the extent of the damage at the interfaces following the VDE simulations.

Failure Analysis of Beryllium Tile Assemblies Following High Heat Flux Testing for the ITER Program

Introduction

Throughout the course of the ITER project several different tile design schemes were evaluated by the joint ITER team. The ITER team consisted of scientists and engineers from Japan, Europe, Russia, and the United States concerned with all aspects of designing and producing a tokomak fusion reactor. Within that framework, Task Group T221 was concerned specifically with the design and production of plasma facing components (PFC). The plasma facing components (PFC) or armor candidate materials consisted of a carbon/carbon composite, beryllium and tungsten. The heat sink candidate materials consisted of ODS copper alloy (Glidcop), CuCrZr copper alloy (Elbrador), and CuBeNi (Hycon-3). The selection of the candidates was based upon several different parameters including: plasma erosion resistance, propensity for tritium retention, thermal conductivity, transmutation products, and location within the reactor. ITER heat fluxes were set at long-pulse heat loads of 5-15 MW/m^2 for the first wall, baffle, limiter, and dome sections and 20 MW/m^2 for the divertor section. The United States T221 Task Group was to work on bonding beryllium and tungsten to one of the candidate heat sink materials. For the armor plate, beryllium was to be considered for the first wall, baffle, limiter, and dome sections and tungsten for the baffle, dome, and divertor sections. For the heat sink material, the three material candidates mentioned above were considered initially but later reduced to CuCrZr because of concerns with thermal and radiation stability at service conditions. Following the assembly process, the tiles were required to meet the physical and mechanical property requirements and remain metallurgically stable following ITER service conditions. This paper describes the development and high heat flux testing of the beryllium-copper alloy tiles for the ITER reactor.

Beryllium Bonding: General Concerns

Initially there were several methods of bonding being considered including electroforming copper on beryllium, inertia welding, brazing, and diffusion bonding. The only restriction was the use of silver-based filler metals, based on the long-lived transmutation products resulting from silver being bombarded by high energy neutrons [1]. These filler alloys had already been successfully used to join beryllium to copper in the pre-ITER years. One such tile assembly was tested in the Joint European Torus reactor (JET) at heat loads to 14 MW/m^2 for 1000 cycles [2] using the silver-based filler metal CuSil ABA (27.5Cu, 13In, and 1.25Ti).

The primary consideration for joining process development was material compatibility. Most elements form brittle intermetallic compounds with beryllium. Intermetallic formation at the interface could lead to premature failure during service. This problem could be solved by using one or more of the elements which do not form intermetallic compounds with beryllium such as silver, aluminum, germanium, and silicon. However, silver was unacceptable and two of the remaining elements do not make good structural materials. The second concern was the handling of residual stresses at the interface during cool-down from the bonding cycle. That is, thermal expansion differences between the beryllium (15.5×10^{-6} K^{-1} to 200°C) and the copper alloy (18.0×10^{-6} K^{-1} to 200°C) will produce stresses near the yield point of the beryllium if a judicious selection of materials or design was not made. The problem of residual stresses was one factor that led the US ITER team to explore joining methods that could be used at lower temperatures. Lower process temperatures would equate to lower residual stresses following fabrication. The other techniques used to accommodate residual stresses included castellations in the beryllium tiles and compliant layers between the beryllium and the copper alloy.

Beryllium-Copper Bonding: Brazing

Brazing was the first bonding process considered for the tile assembly production. Earlier successes with brazing processes held possibilities provided the correct set of materials and processing parameters could be developed. The challenge was to bond a beryllium tile to a CuCrZr (Elbrador) heat sink. The obvious choices (excluding silver) for filler metals were Al, Al-Si, and Al-Ge. All were compatible with beryllium as mentioned above. Unfortunately, all are not compatible with copper. Al-Cu forms several intermetallics which are not acceptable for structural applications. The problem was keeping the aluminum filler metal from reacting with the copper alloy. The solution was a diffusion barrier between the two metals. The question of which material to use for a barrier between these two metals was answered by an explosive bonding company in the United States (Northwest Technical Industries, NTI). For several years they had been fabricating a copper cladding on aluminum plate using a thin (0.25mm) commercially pure titanium sheet as a barrier. The reaction between aluminum and titanium was insignificant at their processing parameters. A contract was placed with NTI to provide a similar clad assembly consisting of 1100-Al bonded to CuCrZr with a 0.25mm layer of titanium. A 0.13mm thick layer of titanium was later tried and discarded because the thinner sheet ripped during bonding. With the copper alloy-aluminum interface problem being solved for the time being, our efforts were directed towards the beryllium-aluminum interface. Beryllium forms a tenacious oxide at room temperature which inhibits wetting by braze alloys. To handle this problem, several years earlier Lawrence Livermore Laboratories (Livermore, CA) had removed the beryllium oxide by ion-sputtering and, in situ, coated the surface with aluminum [3]. Excellent adherence of the aluminum to the beryllium surface was reported by the investigators. This was the process used in our study. The final stage was to bond the beryllium-aluminum and aluminum-CuCrZr alloy using the candidate filler metals. The aluminum serves two purposes. First, it is easier to bond the armor to the heat sink with this transitional material and second, it acts as a compliant layer to reduce the residual stresses created by the thermal expansion mismatch between copper and beryllium. Tensile tests on small-bonded samples processed in this fashion failed in the aluminum compliant layer at the theoretical fracture strength for annealed unalloyed aluminum (115-120 MPa).

The tile assembly was brazed in a hot isostatic press (HIP) furnace using Al-Si and Al-Ge filler metal. The HIP parameters were 625°C for 60m at 105 Mpa. Examination of the Al-Si bond using energy dispersion x-ray mapping (EDX) and scanning electron microscopy (SEM) indicated that the interface had completely disappeared and the silicon had diffused throughout the entire volume of aluminum. This was evidenced by silicon-rich precipitates at the beryllium-aluminum and the aluminum-titanium interfaces. The Al-Ge bond was less acceptable. The germanium had diffused to the beryllium-aluminum interface and formed a continuous germanium-rich phase which produced a very low-strength, brittle bond. The Al-Si filler metal was chosen as the prime candidate for this bonding process. The completed tile assembly is shown in Figure 1. Details of this process were described in an earlier report. [4]

Beryllium-Copper Bonding: Diffusion

As in the case with the brazed assembly, the assembly began with ion-sputtered aluminum (100μm) on beryllium as the armor section and the explosively bonded aluminum to CuCrZr as the heat sink section. To improve on the bond strength achieved in the brazed assembly would require a compliant layer that was both stronger than aluminum and compatible with beryllium. The solution was a composite alloy produced by Brush-Wellman called AlBeMet. Several variants of this composite are available. The one chosen for this application was AlBeMet-150 which consisted of 50% (by weight) Be and 50% (by weight) Al. Tests on this composite by the manufacturer gave a yield strength of 500 MPa.

Several process iterations using the 1100-aluminum alloy and the AlBeMet-150 composite as the compliant layer were attempted and eventually chosen as candidates for further studies. This down-selecting was based on tensile test data and subsequent fracture analysis. Initially, the aluminum-coated beryllium was diffusion bonded to the copper-titanium-aluminum assembly using a hot isostatic pressure (HIP) bonding technique. The bonding parameters were 625°C for 60m at a pressure of 105 MPa. The interface was lined with aluminum oxide particles. In spite of the high pressures produced by the HIP process, break-up and dispersion of the oxide film was difficult and incomplete. Further it wasn't clear that diffusion was optimized by the aluminum-aluminum interface. The solution was to clean and ion-sputter a thin film (100nm) of copper on each aluminum surface prior to bonding. The aluminum oxide was removed and the diffusion was enhanced by the concentration gradient created by the copper-aluminum interface. The copper concentration was less than required for intermetallic formation which would weaken the bond strength. Bond strengths on this assembly and assemblies with the AlBeMet-150 provided excellent results. Details of this process were discussed in an earlier report. [5]

EBTS Test Sequence for the Divertor Mockups (PB-4 and PB-7)

The two assemblies representing the two bonding methods were tested simultaneously. High heat flux tests using the EBTS facility were completed on the two Be/Cu small-scale divertor mockups (PB-7, PB-4). Results show an improved thermal fatigue resistance by using a compliant interlayer material. Both mockups had identical geometry and used S-65C beryllium. Both mockups used Be tiles that were castellated into 4 mm x 4 mm square pillars (total of 64 pillars). The dimensions of the mockup tile assembly are shown in Figure 2. Half of these pillars were 5 mm tall, and the other half were 10 mm tall. The castellation cuts run all the way through the beryllium, through the compliant interlayer, and 0.5 mm into the Cu-alloy heat sink base. The mockups were instrumented with thermocouples located at the Cu/Al interface and actively water cooled. The two mockups were mounted side-by-side using a common water inlet and outlet manifold. Water flow conditions were 15 m/s, 1MPa, 20°C. The thermal simulations in Phase I were the requirements set by the ITER joint central committee for first wall applications. The subsequent phases were conducted to test the tile assemblies under a simulated vertical disruption event.

The HHF testing was accomplished in the following manner. Thermal fatigue cycles were applied with 10 seconds of heating, followed by 10 seconds of cooling. This was sufficient to reach 90% of steady-state conditions. Testing was then organized into four phases.

In Phase I, a 5 MW/m^2 heat load was applied to all of the tiles (e.g., pillars) for 1000 cycles. Typical temperatures were: 180°C at the copper interface, 500°C at the surface of the 5 mm thick Be, and 800°C at the surface of the 10 mm thick Be. No damage to any tiles could be seen. The mockups following this test sequence are shown in Figures 3 for PB-7. The heat staining on the longer tiles is evident in the photographs and a direct result of the higher temperatures achieved as the distance from the cooling source increases. The diffusion bonded assembly (PB-4) exhibited similar heat tinting with no visible damage to the tiles. At this phase of testing, the IR measurements indicated uniform bonding throughout the mock-ups.

In Phase II, an additional 870 cycles was applied to the brazed mockup (PB-7) at 5 MW/m^2 with no obvious damage (total = 1870 cycles). At the same time (during Phase-II) we also heated only the 5 mm thick tiles on the diffusion bonded mockup (PB-4) at 10 MW/m^2. Surface temperatures (monitored by IR) were in the range of 800-900°C at the end of 10 seconds of heating. No additional damage to any tiles could be seen after these cycles. Attempts to heat the 5 mm thick tiles on PB-4 higher than 7 MW/m^2 failed because of a strange "tiger striping" pattern of parallel hot lines that appeared on the Be surface. Apparently, the wave-like morphology created in the titanium by the explosion bonding technique, caused these surface temperature ripples because of the poor thermal conductivity in the titanium.

In Phase III, simulated Vertical Disruption Event (VDE) heat pulses were applied to both tile assemblies. Estimates of the applied energy pulse were 200-250 MJ/m^2 over a time pulse of 0.5 seconds. In this phase of testing, 10 of these pulses to 1/2 of the 5 mm thick tiles and 1/2 of the 10 mm thick tiles were applied to each mockup. The brazed tile assembly (PB-7) following the VDE shots is shown in Figures 4. As evidenced in the photograph, each VDE shot produced significant melting. In some cases, almost all of the 5 mm and some 10 mm thick pillars were completely melted. The beryllium re-solidified both on top of the pillars, and also along the sides of the mockup as smooth, dome-like shapes. Sometimes, the melted surface bridged across 2-3 pillars. Despite this severe heating, all of the tiles cooled down rapidly (within a few seconds), which typically indicates good bonding to the water-cooled substrate. All of the viewing windows were rapidly coated with evaporated beryllium, thus preventing the use of the TV camera, IR camera, or IR pyrometers.

Finally, in Phase IV the original parameters used in Phase I were applied, namely, 5 MW/m^2 for 10 seconds on, 10 seconds off. Because of the severe melting damage on PB-4, many of the tiles could not be irradiated. Therefore, on PB-4, we only applied the 5 MW/m^2 to the left half of the mockup (i.e.; 1/2 of the 5 mm thick Be and 1/2 of the 10 mm thick Be). However, on PB-7 mockups, the entire surface (i.e. both 5 and 10 mm thick Be tiles) could be irradiated. After 400 cycles, no additional damage to PB-7 could be seen. However, some tiles on PB-4 appeared to have become detached and were cooling slowly indicating a change in the bond integrity. Finally, after 530 cycles, some tiles on both mockups were clearly detached. Nevertheless, at this time none of the 10 mm thick tiles on PB-7 appeared to have debonded. Testing was stopped after 1000 cycles. Some of the 10 mm thick tiles on PB-7 appear to have debonded at this point. However, there were many tiles, on both PB-4 and PB-7, that still were well attached.

Failure Analysis: Brazed Tile Assembly (PB-7)

The tiles in PB-7 survived the 10 MW/m^2 heat loads with no visible damage to the assembly. At the higher VDE heat loading, although the tiles in this assembly were intact, there were several areas of delamination near the original aluminum-beryllium interface. A crack front is evidenced near the aluminum-beryllium interface (Figure 5, note arrow). Scanning electron microscopy coupled with energy EDX of the fracture region revealed an abundance of silicon-rich (Si), titanium-rich (Ti), and iron-rich (Fe) phases (Figure 6). The actual crack propagated along a planer region defined by the original brazed surface. This fracture region is located by the original aluminum coating seen on the beryllium (25 μm). As beryllium has limited solubility with all elements it is predictable that this interface would act both as a diffusion barrier and a collection site for all elements diffusing to this region. In contrast, at the titanium side of the bond region, a similar analysis shows a continuous layer of titanium aluminides (Figure 7). The combined effect of time, temperature, and elemental constituents resulted in a multitude of phases.

Failure Analysis: Diffusion Bonded Tile Assembly (PB-4)

Inspection of the 5 mm thick tiles on PB-4 that saw 10 MW/m^2 showed some evidence of minor plastic deformation and surface cracking (e.g. classic mud-flat cracking). Following the VDE heat loading, the unmelted tiles showed no damage to the aluminum or the AlBeMet-150 interlayers. Although, some slight extrusion of the PVD aluminum coating into the castellation gap was noted. X-ray mapping indicated a fracture preference along a thin discontinuous layer of silicon-rich particles near the original aluminum-aluminum interface (Figure 8). No additional phases seen in the PB-7 tile assembly were noted. At the titanium side of the bond region, the AlBeMet-150 had chemically reacted with the titanium to form titanium aluminides.

Discussion

The beryllium-aluminum tile assemblies survived the thermal fatigue to 10 MW/m^2 with no evidence of cracking. Higher heat loads developed by the VDE simulations caused severe melting of the beryllium tiles. The time-temperature histories were sufficient to produce diffusion at the interfaces. The net result was the formation of intermetallic compounds coupled with high stresses sufficient to cause failure at the beryllium-aluminum interface. The fracture propagation for the brazed assembly (PB-7) was along a planer region defined by the original brazed surface. The phases found in this general region were predominantly from the Al-Si filler metal and the CP titanium. In this tile assembly, the compliant layer was a combination of aluminum and the Al-Si filler metal. For the diffusion bonded assembly (PB-4), the failure was in the aluminum region formed by the combined 100 μm of aluminum coating on the beryllium and AlBeMet-150. The fracture appears to have propagated along a discontinuous plane of silicon-rich particles defined by the original aluminum-aluminum interface. The titanium interface in both tile assemblies showed evidence of titanium aluminides. No evidence of cracking due to the presence of these aluminides was observed.

Conclusions

1. The brazed (PB-7) and diffusion bonded (PB-4) beryllium tiles survived 1000 cycles at 10 MW/m^2, and nearly 3000 cycles (2870) at 5 MW/m^2, without damage. Both the pure aluminum and the AlBeMet-150 compliant interlayers performed very well.

2. Severe melting occurred when VDE simulated heat loads (200-250 MJ/m^2(0.5s)) were applied to the tile assemblies. The extreme thermal loading produced intermetallic compound formation at the beryllium-aluminum and titanium-aluminum interfaces in both tile assemblies. The intermetallic compound formation coupled with the thermally-induced stresses during cool-down resulted in delamination of the beryllium tiles.

3. Based on these test results, use of 5 mm thick beryllium armor on limiter modules for ITER can be envisioned for use up to 10 MW/m^2.

4. Improvements in these mockups could include:
   (1) use of beryllium brushes or rods to control surface microcracking,
   (2) replacement of the explosion bonding process with PVD coatings,
   (3) minimizing the thickness of the titanium diffusion barrier layer, or replacement with a higher thermal conductivity material,
   (4) minimizing the thickness of the compliant layer to improve thermal performance.

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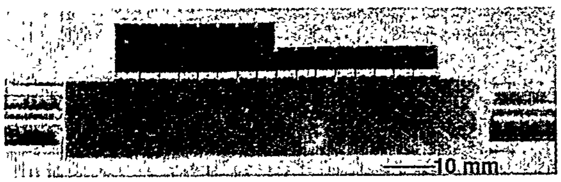


Figure 1

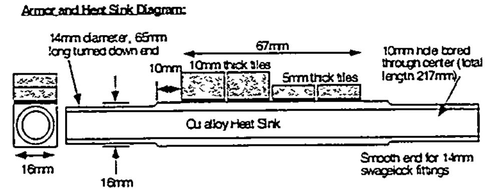


Figure 2

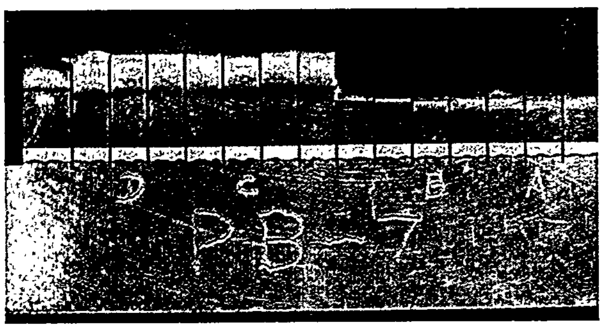
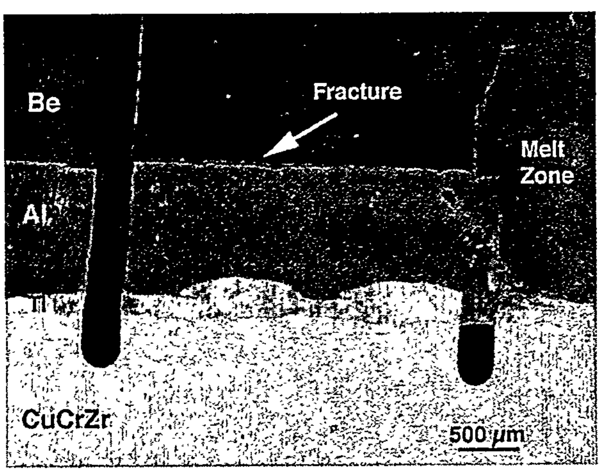


Figure 3


Figure 4



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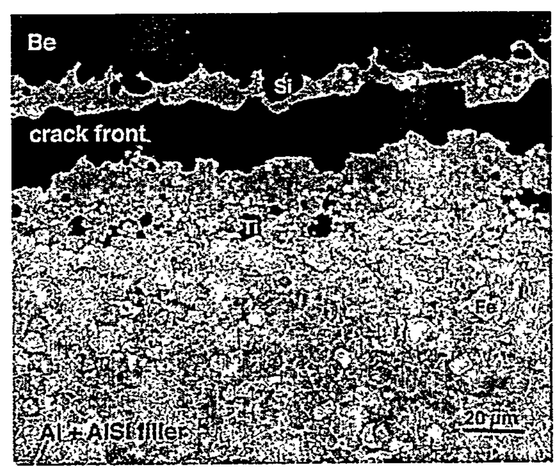

Figure 6

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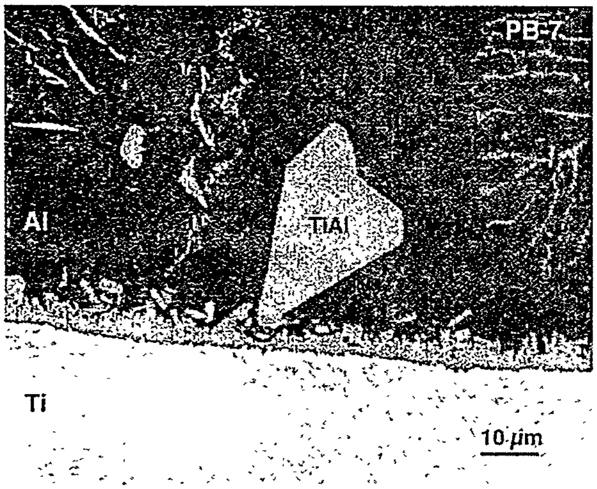

Figure 7

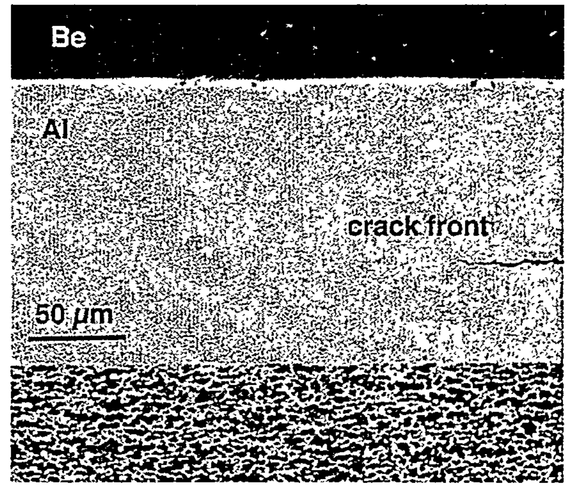

Figure 8

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Figure Captions

Figure 1: Photograph of brazed tile assembly (PB-7) prior to testing in the EBTS facility.

Figure 2: Line drawing showing dimensions of the EBTS high heat flux sample.

Figure 3: Photograph of brazed assembly (PB-7) following heat load exposures to 10 MW/m^2. There was no visible evidence of delamination following this heat load.

Figure 4: Photograph of brazed assembly (PB-7) following heat load exposures to 250 MJ/m^2 (0.5s) to simulate a VDE. The molten beryllium has slumped and in contact with the CuCrZr heat sink.

Figure 5: Photomicrograph of the aluminum/beryllium interface in PB-7 following heat loads to 250 MJ/m^2 (0.5s). A crack is propagating along a planer region near the original beryllium-aluminum interface.

Figure 6: A closer examination of the fracture region in PB-7 following the VDE heat loads. The fracture is located at a planer region defined by the original aluminum-filler metal interface. Several intermetallic compounds are in evidence including: iron-rich (Fe), titanium-rich (Ti), and silicon-rich (Si). The silicon-rich particles are at the beryllium-aluminum interface and only evident using x-ray mapping.

Figure 7: Photomicrograph of the titanium/aluminum interface in PB-7 following heat loads to 250 MJ/m^2 (0.5s). The lighter phase is a continuous layer of TiAl intermetallic.

Figure 8: Photomicrograph of a crack in PB-4 following the VDE heat loading. The crack is propagating along the original aluminum-aluminum diffusion bonded interface.

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