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Assessment of Powder Processing Methods for Production of a Small Modular Reactor Vessel Component Assemblies via Powder Metallurgy-HIP

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Abstract

Under US DOE project DE-NE0008629, the Electric Power Research Institute (EPRI), Nuclear Advanced Manufacturing Research Centre (Nuclear-AMRC), together with engineering support from NuScale Power, have embarked on a project to develop and produce major component assemblies of a small modular reactor vessel at 2/3-scale using novel manufacturing technologies. The research involves the manufacture of upper and lower assemblies of the NuScale Power reactor vessel (Figure 1) using both conventional forging and powder metallurgy-hot isostatic pressing (PM-HIP) technologies, together with electron beam welding to join the component assemblies. (1-4) This paper, which supports the PM-HIP development efforts in the DOE project, will focus on the assessment of powder processing methods for PM-HIP production of the upper and lower reactor vessel heads.

Early research by EPRI has demonstrated that A508 powders can be readily produced via gas atomization, consolidated via HIP, and thermally heat treated to produce acceptable metallurgical properties. The efforts have demonstrated that properties meeting the A508 Grade 3, Class 1 specification can be readily achieved through proper processing and manufacturing controls. (5) This paper explores both powder handling methods and vacuum annealing approaches necessary to achieve acceptable metallurgical properties in large A508 components.

Background

EPRI and the Nuclear-ARMC, together with engineering support from NuScale Power, initiated a project in 2017 to develop and produce major component assemblies of a small modular reactor vessel at 2/3-scale using several novel manufacturing technologies. Two major reactor pressure vessel assemblies (Figure 1) are being produced under the project at 2/3-scale to demonstrate several new technologies including: powder metallurgy-hot isostatic pressing, electron beam welding, and diode laser cladding. An example of a large PM-HIP component produced at a 44% scale is shown in Figure 2.

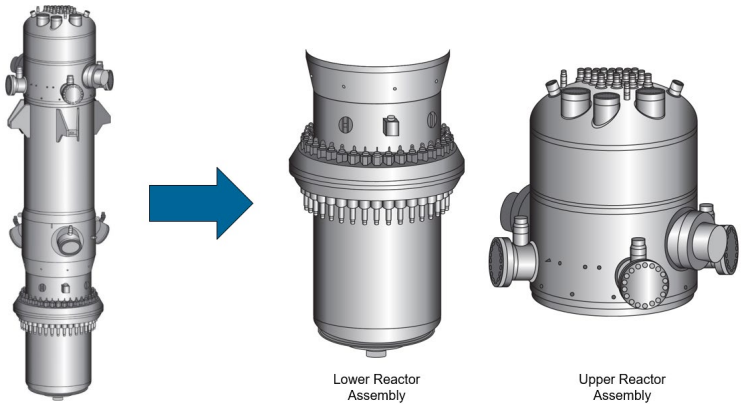

Figure 1. Upper and lower assemblies of the NuScale Power reactor are being assembled at 2/3-scale under DOE project DE-NE0008629.

Figure 2. Upper head of the NuScale Power reactor produced at a 44% scale.

In earlier research by EPRI, together with Carpenter Powder Products, EPRI has demonstrated that A508 powders can be readily produced via gas atomization, consolidated via HIP, and thermally heat treated to produce acceptable metallurgical properties comparable with forged products. The efforts have demonstrated that properties meeting the A508 Grade 3, Class 1 specification can be readily achieved through proper handling, processing and manufacturing controls. (5) This paper explores both powder handling methods and vacuum annealing approaches necessary to achieve acceptable metallurgical properties in large A508 components.

EPRI, Nuclear AMRC and MTC (Manufacturing Technology Centre) initiated the current study to examine the effect of two powder storage conditions on the powder properties and on the HIPed component properties. The MTC performed the environmental storage powder study, characterized the powder properties and encapsulated the powder in HIP canisters, while Nuclear AMRC performed hot isostatic pressing of the canisters and tested the mechanical properties. EPRI performed the microstructural characterization of the HIP'ed canisters and assessed vacuum annealing of the powder as an option to remove residual oxygen just prior to HIP consolidation. The results of this work are described in the following discussion.

Project Objectives

The objectives of this project included:

- Support industry in understanding the effect of storage conditions on oxygen levels and oxide layer thickness for A508 powders.
- Correlation of the relationship between controlled and uncontrolled powder storage conditions; and the properties of A508 powder HIPed components.

- Evaluation of vacuum annealing as a potential method for removal of oxygen from the powder prior to inserting of a capsule into a HIP unit.

Powder Handling Methods

A 250kg container of low alloy A508 steel was forwarded to MTC for characterization and testing of powder handling/storage methods. The powder (heat number of 818651) was produced by Carpenter Powder Products using the gas atomization process. The chemical specification for the powder was given by UNS K12042 (Table 1) and nominal particle size was specified as 53–500 µm.

Table 1. A508 Powder Chemistry—Heat 818651

C	Mn	P	S	Si	Ni	Cr	Mo	V	B	Ti	Al	N	O63
0.15	1.15	0.01	0.007	0.34	0.81	0.1	0.51	0.01	0.001	0.001	0.003	0.03	0.010
0.25max	1.20-1.50	0.025max	0.025max	0.40max	0.40-1.00	0.25max	0.45-0.60	0.05max	0.003max	0.015max	0.025max	NR	NR

The A508 powder was blended and decanted into 4 separate containers and stored under controlled and uncontrolled storage conditions (both described below). At the predefined time periods (0 days, 1 day, 1 week, 2 weeks, 1 month, 3 months, and 6 months), the containers were moved to the encapsulation location where the powders were then sampled and tested using various analysis methods. Once the encapsulation process was completed, the powder containers were resealed and moved back to their original storage locations. The process was repeated for both conditions until the final 6-month time-period elapsed. An additional 2 months were added for the uncontrolled storage condition where the lid was removed from the container and instead replaced by a <11 µm mesh which exposed the powder to the air inside the storage area.

Controlled vs Uncontrolled Conditions

The "controlled storage area" was located in the powder characterization lab (Figure 3) in the main MTC workshop. The set points for the powder characterization lab's temperature and humidity were 18C and 35% relative humidity (rh) respectively. The "uncontrolled storage area" (also shown in Figure 3) was located in a shipping container in the rear yard behind the MTC. The shipping container had only a thermostat which would ensure that the temperature inside the shipping container did not fall below 5C. There was no mechanism to control the humidity. As other items were stored in the same area, the door will have been opened at irregular time periods which will have refreshed the air inside the storage area with fresh air.

Figure 3. Image of MTC's powder characterization laboratory—controlled condition (storage area highlighted in green) and uncontrolled powder storage in shipping container outside.

Powder Sampling and Particle Size Analysis

At predefined time intervals (see above), powder samples (~200 g) were collected from the surface of each container to represent a worse case condition and stored under argon until processing. Samples tested by the MTC were first sent for interstitial analysis, followed by particle size analysis. The remaining powder was then sieved using a Retsch AS200 sieve shaker equipped with a <125 µm mesh to collect the finer sized particles for additional interstitial analysis. Separate samples were collected for XPS analysis. The results are described below.

Oxidation Measurements

The concentrations of interstitial elements present in a A508 powder samples were measured using a LECO ONH836 elemental analyzer. The LECO ONH836 used inert gas fusion (IGF) at 2,000 - 3,000°C to disintegrate the sample. Infrared and thermal conductivity detection were used to measure oxygen, nitrogen and hydrogen levels from the gases emitted. Sieved (<125µm) and unsieved (50-500µm) powder test results are provided below.

53-500µm "Unsieved" Powder Test Results. The interstitial oxygen content for the unsieved 53-500µm A508 powder is presented in Table 2. It is expected that, regardless of storage conditions, the oxygen concentrations of the powder samples would increase as, in the presence of oxygen, the oxide layer thickness will ultimately increase over time. Contrary to those expectations, the oxygen content of powders stored in the controlled condition slightly decreased by 7 ppm, while the oxygen content of the powder samples stored in the uncontrolled condition increased by 2 ppm (Figure 4). This disparity is due to the different powder particle size distributions (PSDs) in each container and that there is a large sample to sample variation in the oxygen content of the powders as the powder PSD is wide in both controlled and uncontrolled powder containers. Nevertheless, *after 6 months there was only a 2ppm difference* between the average oxygen content of the powders stored in either the controlled (72 ppm) or uncontrolled (74 ppm) condition.

Table 2. Oxygen concentration of powders after storing for different times and conditions.

Period	Total delta (days)	Controlled		Uncontrolled		Significance
		Oxygen content (Wt.%)	RSD	Oxygen content (Wt.%)	RSD	P=0.05
Day 0 – before blending	0	0.0087	11.49%	0.0087	11.49%	-
Day 0 – after blending	0	0.0073	11.49%	0.0080	17.50%	No
Day 1	1	0.0088	24.66%	0.0061	2.95%	No
Week 1	8	0.0074	11.36%	0.0078	15.38%	No
Week 2	14	0.0066	9.19%	0.0066	10.30%	No
Month 1	29	0.0073	11.28%	0.0077	10.88%	No
Month 3	91	0.0068	7.31%	0.0075	13.20%	No
Month 6	182	0.0066	13.97%	0.0078	4.49%	No
Month 7	224	-	-	0.0085	1.53%	No
Month 8	252	-	-	0.0086	3.26%	No
Month 6 UO OB	182	0.0052	5.39%	0.0063	4.60%	No
Month 6 UO CB	182	0.0057	2.64%	0.0062	7.56%	No
All periods to 6 months	182	0.0072	10.59%	0.0074	9.77%	No

From months 6 to 8, the lid was removed and opened for the uncontrolled container of powder which resulted in an 8ppm increase in the oxygen content of the powder. This increase in oxygen content

can be attributed to the higher relative humidity the A508 powder was exposed to in the final 2 months. Up to month 6, the relative humidity was approximately 10% relative humidity (rh), while in month 7 and 8, the humidity was on average 78.1%rh and 83.4%rh respectively. It is possible that some of the surface level powder was slightly colder in some areas, sufficiently cold enough for the relative humidity to reach 100 %rh and cause dew formation on the powder accelerating the oxidation process.

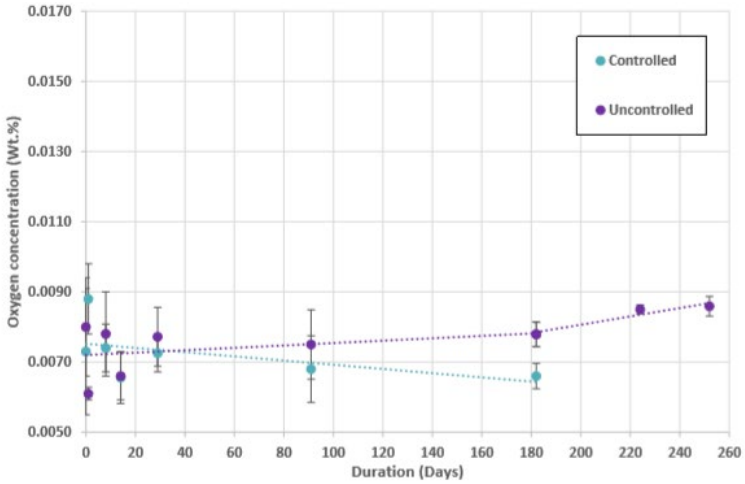

Figure 4. Oxygen concentration of "50-500 µm powders" after storing for different times and conditions

<u><125µm "Sieved" Powder Test Results</u>. The interstitial oxygen content for the sieved powder (<125µm) is presented in Table 3. The oxygen content of powders stored in both storage conditions increase over time with a 12ppm increase in the oxygen content of the powder stored in the controlled condition and 15 ppm increase in oxygen content for the powder stored in the uncontrolled condition. As the PSD is narrower, the sample to sample variability in oxygen content decreased for the <125µm sieved powder samples. The data was also partially normalized for a specific powder cut so there was reduced variability between the PSDs of each container and therefore, oxygen content.

Table 3. Oxygen concentration of sieved powders (125µm) after storing for different times and conditions.

Period	Total delta (days)	Controlled		Uncontrolled		Significance
		Oxygen content (Wt.%)	RSD	Oxygen content (Wt.%)	RSD	P=0.05
Day 0 – before blending	0	0.0134	2.91%	0.0134	2.91%	-
Day 0 – after blending	0	0.0123	1.38%	0.0118	2.88%	No
Day 1	1	0.0125	4.08%	0.0116	6.96%	No
Week 1	8	0.0124	4.19%	0.0127	1.10%	No
Week 2	14	0.0119	1.60%	0.0127	2.28%	Yes
Month 1	29	0.0138	1.16%	0.0135	4.16%	No
Month 3	91	0.0132	1.29%	0.0131	1.15%	No
Month 6	182	0.0135	0.51%	0.0132	3.48%	No
Month 7	224	-	-	0.0135	1.33%	-
Month 8	252	-	-	0.0147	0.39%	-
Month 6 UO OB	182	0.0125	2.72%	0.0134	2.69%	Yes
Month 6 UO CB	182	0.0120	3.99%	0.0138	4.64%	No
All periods to 6 months	182	0.0128	5.39%	0.0127	5.49%	No

As seen with the unsieved 53–500µm A508 powder, between months 6 and 8 for A508 powder stored in the uncontrolled condition, the oxygen content of the A508 powder increased from 132 ppm to

147 ppm. For the same reason, this appeared to be attributed to the significantly higher relative humidity that the A508 powder was exposed to. The oxygen content increase for the <125µm powder was also higher (15 ppm rise) than for the 53 – 500µm powder (8 ppm rise—Figure 5), this highlights how the finer particles oxidize at a higher rate than the coarser particles.

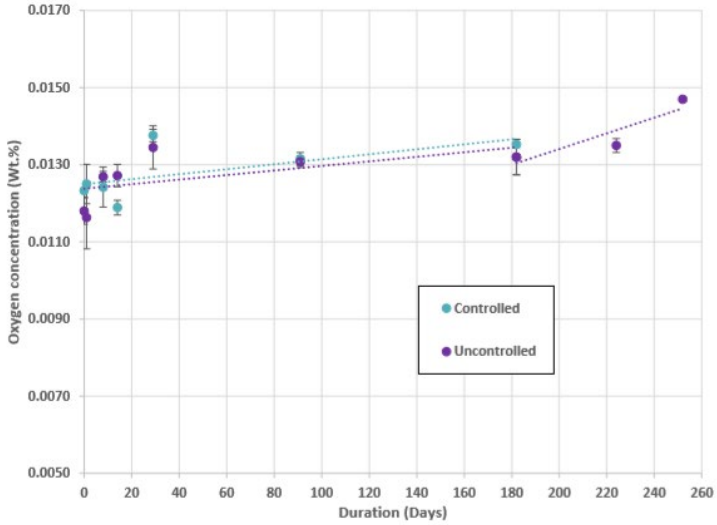

Figure 5. Oxygen concentration of "<125 µm sieved powders" after storing for different times and conditions

X-Ray Spectroscopy

The x-ray spectrographic test results are provided in Table 4 which show the atomic percent depth for the UNS K12042 powder (53–500 µm). Between day 0 and month 6 in the controlled condition, the A508 powders estimated mean oxide thickness increased by 5 nm and the maximum thickness oxide increased by 8 nm with a 1.8% proportional increase in the oxide profile area. Between months 6 to 8 where the uncontrolled powder storage container bag was open and lid removed, the estimated mean oxide thickness increased by 4 nm and maximum thickness oxide thickness increased 12 nm with a 2.2% proportional increase in the oxide profile area.

Table 4. X-ray photospectroscopy results for A508 powders stored under different times and conditions.

Powder ID	Estimated mean thickness (nm)	Estimated maximum thickness (nm)	Estimated area (nm %)
C 0D O CB	18	27	680
C 6M O CB	23	35	692
U 6M O CB	29	45	917
U 8M O OB	33	57	937

Powder Consolidation Via Hot Isostatic Pressing

As noted earlier, MTC performed the environmental storage powder characterization and tested the properties of the powders under different storage conditions. MTC also encapsulated the powders into small cylindrical canisters (100mm diameter x 200mm long) for both the controlled and uncontrolled powder conditions and forwarded them to Nuclear AMRC for HIP. The HIP parameters and heat treatment schedules employed for all powder canisters were:

- HIP parameters: 1120°C, 103MPa, 4 hours

- H/T schedule: Solution anneal (1120°C/2 hours WQ), Normalize (870°C/10 hours WQ) and Temper (650°C/10 hours AC)

Following HIP, each canister was sectioned and mechanical properties were assessed. Mechanical properties included: 1) Charpy V-notch toughness and tensile testing. In addition, oxygen measurements were collected for each consolidated canister.

Charpy V-notch testing was performed along the longitudinal and transverse directions for each canister. The results are shown in Figures 6 and 7 respectively. Very consistent toughness properties were observed under both controlled and uncontrolled conditions out to 3 months of exposure with "no appreciable drop-off" in toughness properties. Toughness on the order of 100-110J (74-81ft-lbs) was measured for each exposure condition out to 3 months duration. At the six-month interval, some slight drop in toughness was observed, but even at this duration, good toughness (on the order of 85-90J (63-66ft-lbs) was still observed. These results suggest that powders can be atomized and stored for reasonable periods of time before being consolidated in a component.

Tensile properties were also measured for the controlled and uncontrolled conditions. Figure 8 provides the ultimate tensile strength (UTS) where no drop was observed even out to an 8-month duration. Similar results were also observed for under tensile proof test conditions, but are not included in this document.

The Nuclear AMRC also performed oxygen measurements for each of the consolidated capsules. Oxygen measurements were obtained at the edge of each capsule (not shown herein) and at the center of each (Figure 9). Oxygen levels were shown to be highest at the 3-, 6-, and 8-month intervals corresponding well with the Charpy-V notch results described above. Oxygen levels were on the order of 120-130ppm at these durations for measurements taken near the edge of the consolidated canisters, while it was just slightly lower (120ppm) at the billet center. The results suggest that low oxygen levels can be readily attained provided the original powder oxygen level was low.

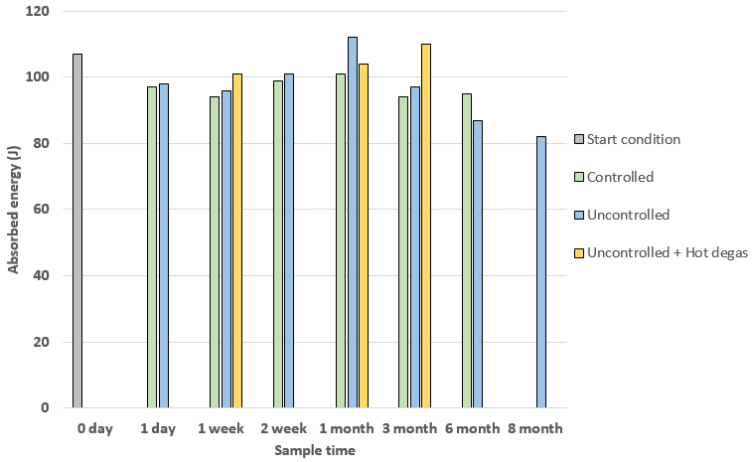

Figure 6. Comparison of the longitudinal Charpy toughness for PM/HIP A508 billets stored under differing conditions based on exposure time

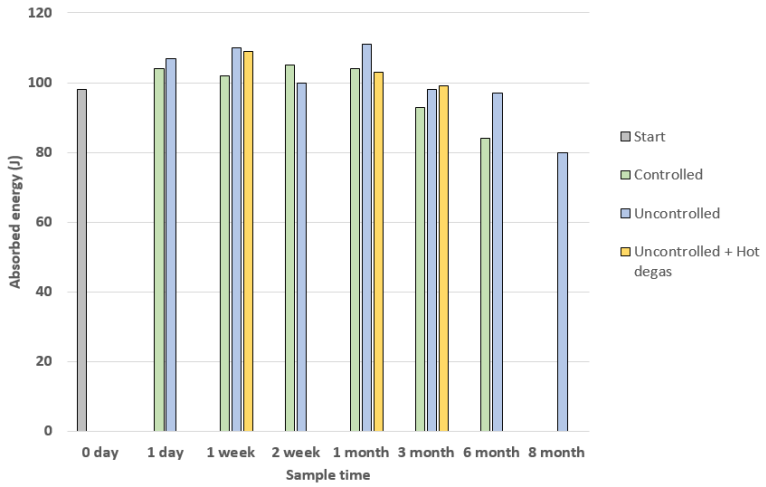

Figure 7. Comparison of the transverse Charpy toughness for PM/HIP A508 billets stored under differing conditions based on exposure time

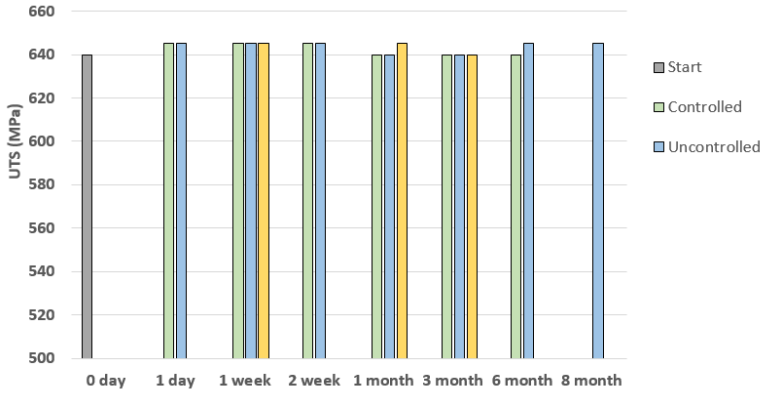

Figure 8. Plot of UTS for the controlled/uncontrolled A508 storage conditions after HIP consolidation.

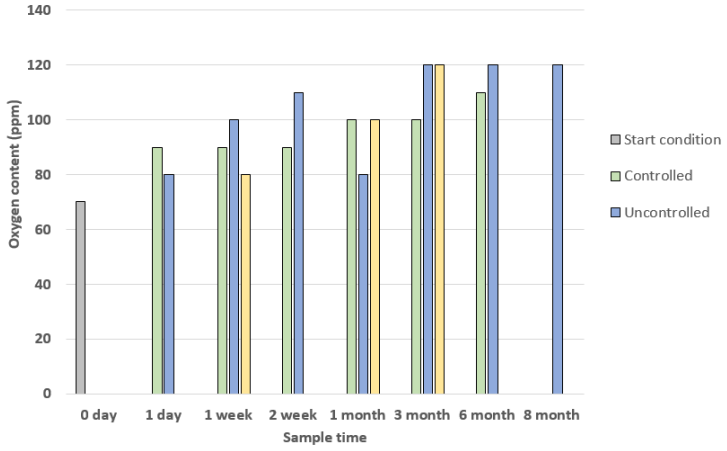

Figure 9. Plot of the oxygen content (billet center) for PM/HIP A508 billets as a function of storage time and conditions

Vacuum Annealing of Powders

A third part of this study focused on vacuum annealing of A508 powders to determine if this method could be employed to "clean" powders (ie., remove residual oxygen) just prior to degassing and HIP consolidation of a component. The study included: 1) fabrication of four 150x150x150mm cubical capsules, 2) open air filling of each capsule with A508 powder, 3) cold degassing of each capsule and backfilling each with argon, 4) and then a high vacuum/high temperature vacuum anneal heat treatment of each capsule for 4 hours each. Vacuum annealing temperatures of 600C, 700C, 800C, and 900C were utilized for the four different capsules. This part of the study was conducted jointly by Synertech-PM and EPRI.

Following the vacuum anneal, each capsule was hot out-gassed at 120C to remove the argon and then each capsule was sealed via crimping/welding of the fill tube. The capsules were then HIP'ed at 1120C (2050F) at 100MPa (15ksi) for four hours and allowed to cool in the HIP furnace. Next, the HIP consolidated capsules were solution annealed at 1120C (2050F) for 2 hours, water quenched, normalized at 900C (1650F) for 10 hours, water quenched, and finally tempered at 650C (1200F) for 10 hours and allowed to air cool. Three Charpy V-notch toughness specimens and two tensile specimens were removed from each capsule and mechanically tested. The results of the testing are provided in Tables 5 and 6.

Table 5. Room temperature tensile results for four sets of tensile tests at 600C, 700C, 800C, and 900C.

Vacuum Annealing Temperature (C)	Tensile Strength- - MPA (ksi)	Yield Strength- - MPA (ksi)	Elongation in 4D (%)	Reduction of Area (%)
600	639 (92.8)	516 (74.8)	24.5	68
700	628 (91.1)	501 (72.7)	25.8	72.5
800	635 (92.1)	511 (74.1)	26	70
900	633 (91.9)	508 (73.7)	24.5	67.5

Table 6. Room temperature (20C) Charpy impact test results for four sets of toughness tests at 600C, 700C, 800C, and 900C.

Vacuum Annealing Temperature (C)	**Joules**	**Ft-lbs**	**Lateral Expansion (inch)**	**Shear (%)**
600-1	101.5	75.0	0.073	80
600-2	80.0	59.0	0.053	70
600-3	115.0	85.0	0.061	90
600-Average	**99.0**	**73.0**	**---**	**---**
700-1	133.5	73.0	0.082	90
700-2	125.5	98.5	0.08	90
700-3	118.5	92.5	0.072	90
700-Average	**126.0**	**87.5**	**---**	**---**
800-1	124.5	92.0	0.085	90
800-2	133.0	98.0	0.091	100
800-3	122.0	90.0	0.09	90
800-Average	**127.0**	**93.5**	**---**	**---**
900-1	66.5	49.0	0.054	60
900-2	40.0	29.5	0.036	30
900-3	63.5	47.0	0.047	60
900-Average	**57.0**	**42.0**	**---**	**---**

The tensile results were shown to be consistent across all four capsules which were exposed to different vacuum annealing conditions. Charpy V-notch results determined that 700C and 800C provided excellent results (126- and 127J (ave.) respectively), while the 600C test (99 joules) appeared to be too low to achieve proper "cleaning" of the powder and the 900C test (57 joules) appeared to be over the Ac3 critical temperature of the material resulting in inferior toughness. Further testing is required to determine what the optimum vacuum annealing temperature is for A508 powders, but the current results suggest 700C or 800C both work quite well toward "cleaning" or removing any residual oxygen within the powder.

It should also be pointed out that vacuum annealing of the powder just prior to HIP consolidation resulted in approximately a 27J (20ft-lb ave.) increase over earlier testing where no vacuum annealing was employed prior to HIP. The earlier testing (99J (73ft-lbs ave.) simply utilized open air filling of a capsule, hot degassing at 120C and sealing of the capsule prior to HIP consolidation. Thus, vacuum annealing does offer good improvement toward removal of residual oxygen from the A508 powder.

Summary and Conclusions

The results of the three complementary studies described above suggest the following:

Storage of A508 Powder in Controlled and Uncontrolled Conditions

Testing of A508 powders under controlled and uncontrolled storage conditions suggested that controlled storage practices (temperature, dewpoint controlled) were successful in minimizing oxygen pickup in the powder even out to 6 months of storage time. Oxygen concentrations for 50-500µm A508 powders (controlled and uncontrolled conditions) were measured at <80ppm out to six months storage time, while oxygen concentrations were measured for <125µm powders (controlled and uncontrolled conditions) were up to ~135ppm.

The X-ray photospectrography results suggest that between day 0 and month 6 in the controlled condition, the A508 powder estimated mean thickness and maximum thickness increased by 5 nm and 8 nm respectively with a 1.8% proportional increase in the oxide profile area. Between months 6 to 8 in the A508 powder stored in the uncontrolled condition, the estimated mean and maximum thicknesses increased by 4 nm and 12 nm respectively with a 2.2% proportional increase in the oxide profile area.

HIP Consolidation of Capsules using A508 Powder from Controlled and Uncontrolled Conditions

Very consistent toughness properties were observed under both controlled and uncontrolled storage conditions out to 3 months of exposure with "no appreciable drop-off" in toughness properties. Toughness values on the order of 100-110J (74-81ft-lbs) was measured for each exposure condition out to 3 months duration. At the six-month interval, some slight drop in toughness was observed, but even at this duration, good toughness (on the order of 85-90J (63-66ft-lbs) was still observed. These results suggest that powders can be atomized and stored for reasonable periods of time before being consolidated in a component.

Vacuum Annealing to remove residual oxygen

Test results suggest that the 700°C and 800°C tests were successful in sufficiently reducing oxygen content in the final consolidated capsules resulting in acceptable Charpy toughness results (~127J (93ft-lbs). An improvement of ~27J (20ft-lbs) was observed for the vacuum annealed test coupons over those that were simply exposed to HIP with no vacuum annealing. Thus, vacuum annealing does offer

good improvement toward removal of residual oxygen from the A508 powder and should be considered as an option for achieving consistent properties.

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