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MATERIALS TECHNOLOGY

INFORMATION BRIEF

Title: Experimental Design for Evaluation of Co-extruded Refractory Metal/Nickel Base Superalloy Joints

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Introduction: Prior to the restructuring of the Prometheus Program, the NRPCT was tasked with delivering a nuclear space reactor. Potential NRPCT nuclear space reactor designs for the Prometheus Project required dissimilar materials to be in contact with each other while operating at extreme temperatures under irradiation. As a result of the high reactor core temperatures, refractory metals were the primary candidates for many of the reactor structural and cladding components. They included the tantalum-base alloys ASTAR-811C and Ta-10W, the niobium-base alloy FS-85, and the molybdenum base alloys Moly 41-47.5Rhenium. The refractory metals were to be joined to candidate nickel base alloys such as Haynes 230, Alloy 617, or Nimonic PE 16 either within the core if the nickel-base alloys were ultimately selected to form the outer core barrel, or at a location exterior to the core if the nickel-base alloys were limited to components exterior to the core.

To support the need for dissimilar metal joints in the Prometheus Project, a co-extrusion experiment was proposed. There are several potential methods for the formation of dissimilar metal joints, including explosive bonding, friction stir welding, plasma spray, inertia welding, HIP, and co-extrusion. Most of these joining methods are not viable options because they result in the immediate formation of brittle intermetallics. Upon cooling, intermetallics form in the weld fusion zone between the joined metals. Because brittle intermetallics do not form during the initial bonding process associated with HIP, co-extrusion, and explosive bonding, these three joining procedures are preferred for forming dissimilar metal joints.

In reference to a Westinghouse Astronuclear Laboratory report done under a NASA sponsored program[1], joints that were fabricated between similar materials via explosive bonding had strengths that were directly affected by the width of the diffusion barrier. It was determined that the diffusion zone should not exceed a critical thickness (0.0005 in.). A diffusion barrier that exceeded this thickness would likely fail. The joint fabrication method must therefore mechanically bond the two materials causing little or no interdiffusion upon formation. Co-extrusion fits this description since it forms a mechanical joint between two materials by using heat and pressure. The two materials to be extruded are first assembled and sealed within a co-extrusion billet which is subsequently heated and then extruded through a die. For a production application, once the joint is formed, it is dejacketed to remove the outer canister. The remaining piece consists of two materials bonded together with a thin diffusion barrier. Therefore, the long-term stability of the joint is determined primarily by the kinetics of interdiffusion reaction between the two materials. An experimental design for co-extrusion of refractory metals and nickel-based superalloys was developed to evaluate this joining process and determine the long-term stability of the joints.

Experimental: Contents of the Co-Extrusion Billet:

To support the need for dissimilar metal joints for the Project Prometheus space program, a co-extrusion experiment was proposed. This experiment was to establish the optimum conditions for formation of joints between refractory metals and nickel-base superalloys. The proposed sample configuration for this experiment is listed in Table 1. The details herein describe the work that was planned to occur prior to termination of the NR effort to deliver a space reactor.

	Rod	Tube
Configuration 1	Ta-10W	Haynes 230
Configuration 2	FS-85	Haynes 230
Configuration 3	ASTAR-811C	Haynes 230
Configuration 4	Mo-47Re	Haynes 230

Table 1. Proposed sample configurations for rod and tubular stock billet

Figure 1 depicts the billet design for rods and tubular forms. As shown in Figure 1, the refractory metal alloys and nickel-based superalloys should have been tightly fit together upon assembly; the gap between the materials should have been minimized. Refractory metal foil (typically tantalum or molybdenum) should have encased the superalloy and the refractory metal to prevent contamination from the steel canister during the extrusion process. The nominal chemistries of the candidate refractory metal alloys and nickel-based superalloys that were being evaluated by the NRPCT for the Prometheus Project are listed in Tables 2 and 3.

Weight Percent	ASTAR-811C	FS-85	Ta-10W	Mo-47.5Re
Ta	90.28	27	90	-
Mo	-	-	-	52.5
Nb	-	62	-	-
Re	1	-	-	47.5
W	8	10	10	-
Hf	0.7	-	-	-
C	0.03	-	-	-
Zr	-	1	-	-

Table 2. Nominal Chemistry of Refractory Metal Alloys

Despite their similar chemical compositions, ASTAR-811C and Ta-10W were added into the matrix due to their carbon content (ASTAR-811C) or lack thereof (Ta-10W).

For simplicity, only one type of superalloy was used in the matrix. The chemistry for Haynes 230 is listed in Table 3.

Element	Weight Percent
Ni	52.69
Cr	22.00
W	14
Co	5.00
Fe	3.00
Mo	2
Mn	0.50
Si	0.40
Al	0.30
C	0.10
B	0.015

Table 3. Nominal Chemistry of Haynes 230

Assembly of the Billet:

The samples should have been pickled and annealed prior to the experiment. Surface finishes for these materials were to be approximately 125 RA. The proposed annealing temperatures for the refractory metals and nickel base superalloy are listed in Table 4. Each of these materials should have recrystallized at these temperatures in approximately one hour.

Alloy	Annealing Temperature
ASTAR-811C	1650 °C
FS-85	1500 °C
Ta-10W	1650 °C
Mo-47.5Re	1650 °C
Haynes 230	1200 °C

Table 4. Annealing Temperatures

No welding of the contents inside the canisters should have been necessary. The only welding concern would have been the stainless steel canister holding the refractory metal alloy/superalloy contents. The canister would have been TIG-welded together in air; vacuum would not have been necessary for this assembly.

To minimize oxygen and nitrogen contamination of the refractory metal pieces, the inner chamber would have been evacuated through a pinch valve. The pinch valve is shown in Figure 1. This valve would have allowed a vacuum to be drawn inside the container. Once the desired pressure of 10^{-5} torr was reached, the billet would have been vacuum sealed by pinching off the valve. A pressure greater than 10^{-5} torr would have been unacceptable because oxygen and nitrogen contamination could have resulted at these higher pressures.

The extrusion temperatures would have been in the range of 970 °C-1070 °C (1800-1950 °F). The lower temperature would have been used due to the melting point of the steel canister (T_m = 1538 °C). The reduction ratios would have been on the order of 5-6:1, as determined previously by Wright-Patterson Air Force Base in Dayton, Ohio.

De-cladding of the material would have been performed to remove the outer steel canister. The de-cladding process would have required machining off the majority of the steel canister and then pickling the remaining contents in acid. The pickling process would have needed to be precise to prevent acid attack of the contents inside. The de-cladding procedure would have required the canister to be immersed in a bath of HCl, however, this process could have been modified for improvement.

At the time this report was written, the recommended vendor to fabricate the billet was Pittsburgh Materials Technology (Large, PA), and the preferred vendor to co-extrude the billet was Wright Patterson Air Force Base.

Proposed Results and Analysis:

The definition of a successful bond is one that has limited porosity or any other major defects in the bond area and maintains its strength along the entire bond length. The design criteria for this work were that the joint would be a metallurgical bond and that it be machinable in the as-extruded condition.

The extruded materials would have then been sectioned to analyze the transverse and longitudinal directions. One extrusion should have been able to provide at least 30 specimens. Five to seven specimens of each configuration should have been made to analyze the as-extruded condition as a control for the thermal exposures. A minimum of 24 specimens of each configuration would have been prepared for thermal exposure (Table 5). This would have allowed two samples per configuration for each thermal exposure. The size of the samples was currently undetermined. The initial size of the canister samples would have depended upon the sample size needed for Vickers hardness and tensile testing. The vendors would have needed to be consulted on this issue.

Temp (°C)	*Dwell Times (hrs)*			
760	*100 (2 samples)*	*1000 (2 samples)*	*5000 (2 samples)*	*10000 (2 samples)*
815	*100 (2 samples)*	*1000 (2 samples)*	*5000 (2 samples)*	*10000 (2 samples)*
870	*100 (2 samples)*	*1000 (2 samples)*	*5000 (2 samples)*	*10000 (2 samples)*

Table 5: Thermal exposure dwell times and temperatures for each configuration

The final extruded product would have been inspected for delaminated joints. This inspection, to have been performed via metallography, would have determined the length of the bond area. The goal was to have a successful joint 1) with a bond area that was significantly less than the critical thickness of $5x10^{-4}$ inches, 2) that did not have porosity or any other major defects in the bond area, and 3) that had a bond length that maintained its strength. The joints would have undergone Vickers hardness and tensile testing to further evaluate their properties.

The remaining joints would have undergone a series of thermal exposures. Temperatures and times of interest are listed in Table 5.

It is shown in Table 5 that there would have been 12 different conditions for each configuration. The exposed samples would have then been evaluated to document the change in the diffusion zone. The main interests in the diffusion zone were its length and intermetallics. Intermetallics are not evident during the initial joining process, but they tend to form during thermal exposures. Vickers hardness and tensile testing would have been performed on the thermally exposed samples, and these results would have been compared to those from the as-extruded samples.

Joints that did not pass the inspection after extrusion would have undergone failure analysis, including chemistry and SEM analyses. Any other analyses to determine the cause of failure would have been decided upon at a later date.

If failure did occur, temperatures, surface preparations, and microstructures might have needed to be altered from this first run in the experiment. Due to this chance of failure, it would have been necessary that multiple extrusion packs of the same configurations be made. If failure did not occur, it would have been beneficial to change certain parameters to see if bonding still occurred with better results. It was been suggested that three packs of each configuration be assembled, however, this number might have increased due to the success/failure of the billets.

Conclusions: The goal of this experiment was to obtain information on the co-extrusion joining method. Due to the termination of the NR effort to deliver a nuclear space reactor for Project Prometheus, this experiment was not conducted. Each aspect of the joining procedure would have been analyzed, from the preparation of the samples to the temperatures of the thermal exposures. This experiment would have provided a better understanding of the process and requirements necessary to produce a successful joint.

Significance to NR Program: This experiment was deemed necessary for the material design of the reactor and power system for the Prometheus Project. The results of this test would have aided in concluding which joining method should have been used for dissimilar metals.

Future Action: No future action is planned at this time.

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Reference:

1. Evaluation of Refractory/Austenitic Bimetal Combinations" R. W. Buckman, Jr and R. C. Goodspeed WANL - PR-(EE)-004, August 1969 Prepared for NASA-Lewis Research Center Space Power Systems Division Contract NAS 3-7634

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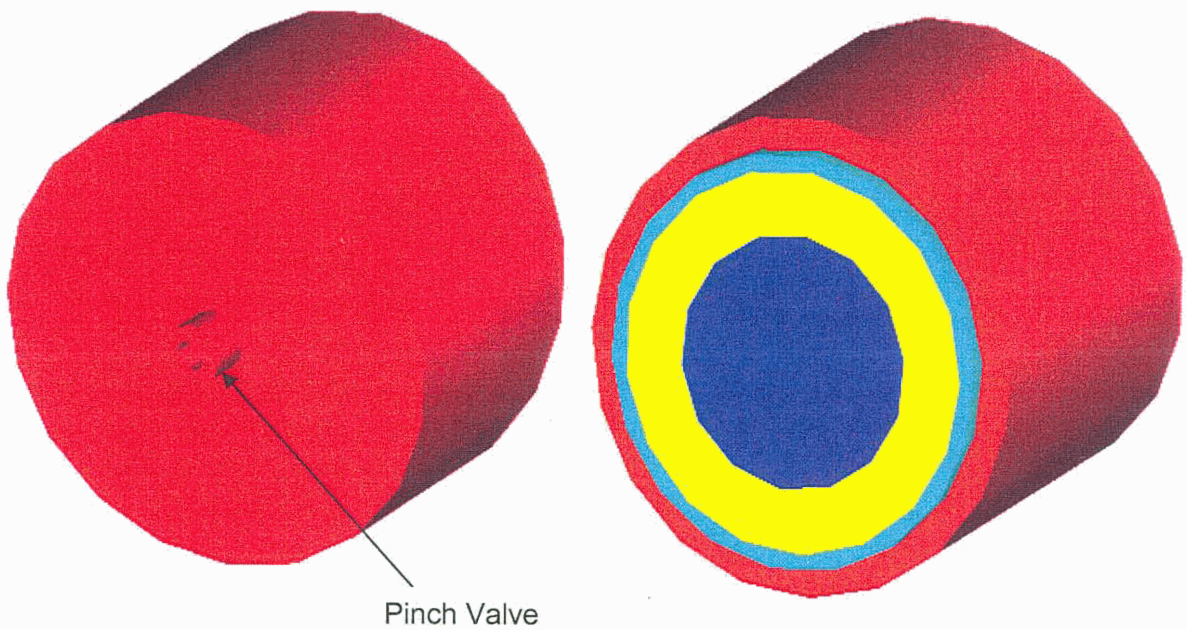

Figure 1. Round Extrusion Pack. Red –Canister, Aqua-Refractory Metal Foil, Yellow-Nickel-Based Superalloy, Blue-Refractory Metal

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