Evaluation of Cold Isostatic Pressing of High-Pressure Thrust Chamber Closeout

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The objective of this project was to investigate new fabrication methods and design techniques that potentially reduce fabrication time and cost, as well as increase rocket thrust chamber life. For these purposes, the authors developed the cold isostatic pressing (CIP) forming method, based on powder metallurgy. Using the CIP forming method, a very compliant closeout was easily obtained while sustaining sufficient bonding strength between the copper liner and the closeout, and providing perfect sealing of coolant channels. A metallurgical test was performed before the fabrication of a trial combustion chamber to determine the forming conditions of the sintered closeout that would meet the required design conditions. The trial combustion chamber was then made to withstand a design combustion pressure of 15 MPa and a thrust level of 10 kN. Combustion tests were conducted at chamber pressures up to 9 MPa for 28 runs to confirm the reliability of the CIP-formed chamber. A preliminary analysis was then made which also demonstrated that a thrust chamber with a compliant, sintered alloy closeout would have a longer life capability than one with a stiffer, electroformed nickel closeout.

Nomenclature

E = elastic modulus
N = number of cycles
P = pressure
S = strength
T = temperature
ΔT = temperature difference
r = width of rib
w = width of coolant channel
α = coefficient of thermal expansion
$\Delta\epsilon$ = strain range
v = Poisson's ratio

Superscripts

$(\ )'$ = strain range caused by thermal expansion across the ligament
$(\ )''$ = strain range caused by thermal expansion between inner shell and closeout

Subscripts

c = combustion
f = failure
j = coolant jacket
p^1 = strain range caused by thermal expansion
tot = total
y = yield

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Introduction

IN order to improve Japan's launching capability for the '90s, a tradeoff system study of the LE-7 booster engine was conducted. A high-pressure, liquid-oxygen/liquid-hydrogen, staged combustion cycle engine system was selected. In the development of the LE-7 engine, the high-pressure thrust chamber fabrication method was considered to be the most critical concern. Various fabrication methods[1] had been investigated in recent years for applicability to the construction of the LE-7 engine.

It was proved that the electroforming method adopted for the Space Shuttle Main Engine (SSME) was not necessarily the most desirable for a long-life engine because of the high stiffness of the electroformed nickel closeout.[2] The objective of this project was to investigate new fabrication methods and

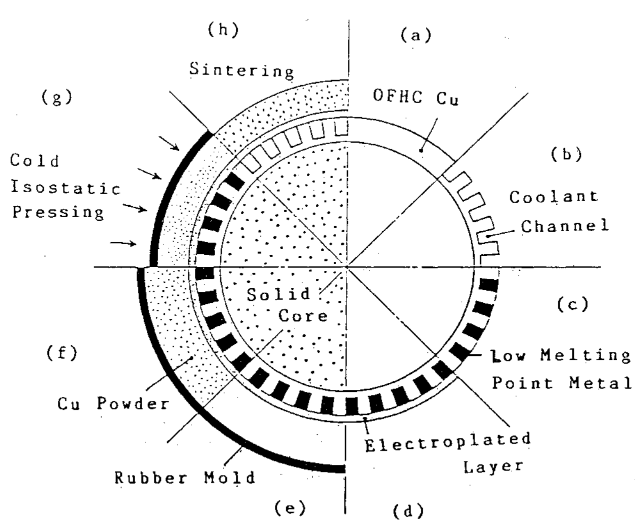


Fig. 1 Fabrication sequence for CIP-formed thrust chamber.

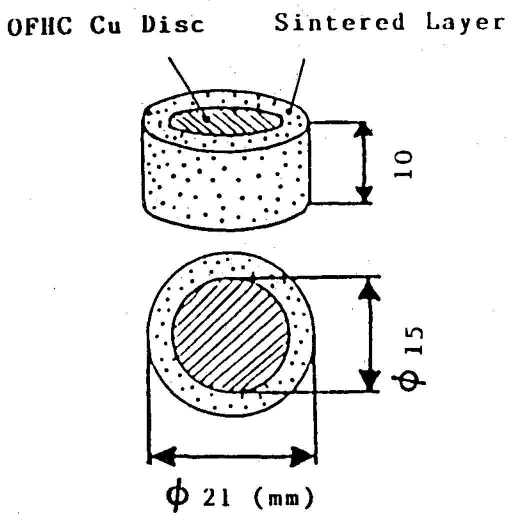


Fig. 2 CIP-formed disk for blanking force test.

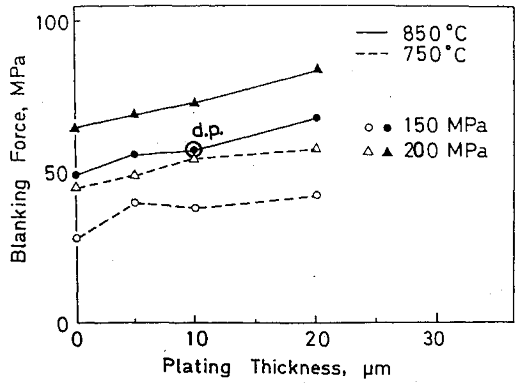


Fig. 3 Results of blanking force tests.

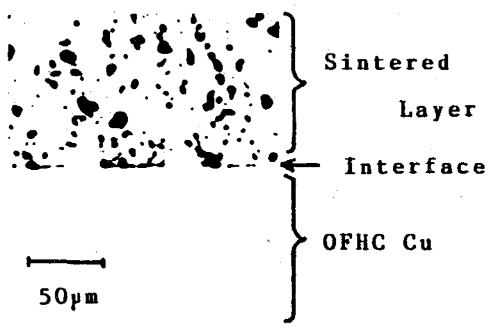


Fig. 4 Microstructure near interface (copper powder and tin-plated OFHC copper).

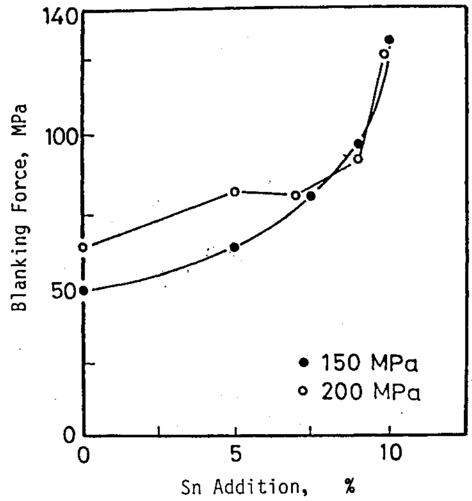


Fig. 5 Effect of tin addition on blanking force.

design techniques which potentially reduce fabrication time and cost, and increase thrust chamber life. The authors developed the cold isostatic pressing (CIP) forming method, based on powder metallurgy.[3]

A brief fabrication sequence of this method, as shown in Fig. 1, begins with a thin copper or copper alloy liner. Coolant channels are milled on the liner. The channels are then temporarily filled with a low-melting-point metal. The liner is thinly plated to seal up the coolant channels. The space between the liner and the flexible mold is evenly filled with copper powder or a powder mixture of copper and other metals. The powder is isostatically compacted against the liner. After cold isostatic pressing, the filling metal is removed. Finally the compact is sintered in a hydrogen gas flow.

A very compliant closeout is obtained easily using this method, while sustaining sufficient bonding strength between the copper liner and the closeout, and also providing perfect sealing of the coolant channels.

Microstructure and Mechanical Properties of a CIP-Formed Interface

The Combination of Copper Powder and Tin-Plated OFHC Copper

An analysis was made prior to the fabrication of a trial combustion chamber to determine the required strength for bonding the OFHC copper liner and the closeout. A trial combustion chamber capable of withstanding a 10-kN thrust, with a combustion pressure of 15 MPa, would theoretically require a bonding strength of 44 MPa.

The forming conditions of the sintered closeout, which would possess the required strength, were studied by using small test pieces, as shown in Fig. 2. The tin-plated, 15-mm-diam, 10-mm-thick OFHC disks, peripherally covered with a copper powder layer about 3 mm thick, were made by cold isostatic pressing at either 150 or 200 MPa. They were then sintered at either 750 or 850°C for 1 h in a hydrogen gas flow.

Figure 3 shows the results of the blanking force test on the sintered specimens as a function of the tin-plate thickness. The blanking force may be safely regarded as one-half of the tensile strength of the bonding interface, as mentioned in the latter results. It is obvious that the blanking force increases with an increase in the plating thickness, sintering temperature, and CIP pressure. However, in the case of a tin-plate thickness of 20 μm, some large residual pores were recognized on the joint interface.

Figure 4 shows the typical microstructure of the specimen interface prepared under the following conditions: CIP pressure of 150 MPa, tin-plate thickness of 10 μm, and sintering temperature of 850°C. Residual pores were confirmed to be closed and no permeability was observed. An EMPA (Electron Probe Microscope Analyzer) analysis showed that the depth of diffusion of the plated tin into the OFHC copper core was about 100 μm, which should imply the formation of a sound joint interface. It is expected that the sintered closeout stiffness could be lowered by controlling the residual porosity, as shown in Fig. 4, which may improve the fatigue property of the combustion chamber.

On the basis of these results, the forming conditions for the sintered closeout of the trial combustion chamber were selected as: CIP pressure, 150 MPa; tin-plate thickness, 10 μm; and hydrogen sintering, 850°C for 1 h. As for the CIP pressure, in a strict sense, the level of 200 MPa should be selected, according to basic research. However, a CIP instrument large enough to fabricate a trial chamber at 200 MPa was unobtainable. Therefore, the level of 150 MPa was decided upon, as it also yields enough strength for the trial chamber.

The Combination of Copper Tin-Mixed Powder and Tin-Plated OFHC Copper

Tests were made to obtain a joined layer having a low stiffness by control of residual porosity and the improved joint strength and controlled microstructure of a copper/tin and copper/silver alloy, instead of copper only. This report treats only the case of copper/tin mixed powder.[4]

Atomized tin powder (particle diameter $<37\mu m$) was added to the copper powder to control the microstructure and strength of the sintered layer, as well as to improve the bonding strength. In this case, the small specimen was isostatically pressed at either 150 or 200 MPa and sintered at 750°C for 1 h and then 850°C for an additional hour in a hydrogen gas flow.

Figure 5 shows the effect of tin addition and CIP pressure on the joint strength of sintered composites. The maximum strength was obtained for specimens with a 10% tin addition, a strength which exceeded the strength of pure wrought copper by about 30%.

Figure 6 shows the microstructure of the copper/tin sintered layer of the specimen with the maximum bonding strength. Spherical pores are seen to be distributed uniformly in the sintered matrix. The porosity near the interface is smaller than that in the inner region of the sintered matrix, presumably due to sintering enhancement by diffusion into the OFHC copper core.

The elastic and plastic properties of the sintered matrix were measured by using dog-bone-type test pieces made by the die-compacting method. The die-compacting pressures were carefully adjusted to obtain the same densities as in the case of CIP forming at 150 and 200 MPa. Figure 7 shows tensile strength and elongation at the fracture plotted against the tin addition. Curves A and B in this figure correspond to the CIP pressures of 150 and 200 MPa, respectively. Figure 8 shows the variation of the elastic modulus of the sintered matrix with a tin addition to the copper powder. The elastic modulus tends to decrease with an increase of tin addition, dropping as low as one-half of that for pure wrought copper.

The structural stiffness is generally evaluated by the multiplication of the modulus of elasticity and the material thickness. The thickness is designed to be inversely proportional to the material strength. As shown in Fig. 8, the modulus of elasticity decreases with an increase of tin addition, while the yield strength of the copper/tin alloy is only slightly affected by the amount of tin addition, as shown in Fig. 7. This fact indicates that the stiffness of the copper/tin alloy can be reduced by increasing the amount of tin addition without spoiling the structural properties.

In a practical sense, dimensional changes brought about by the sintering should be controlled so as to be negligible. Also note that the dimensional changes of these sintered bodies were confirmed to be within a ±10% range under the conditions, as shown in Fig. 9.

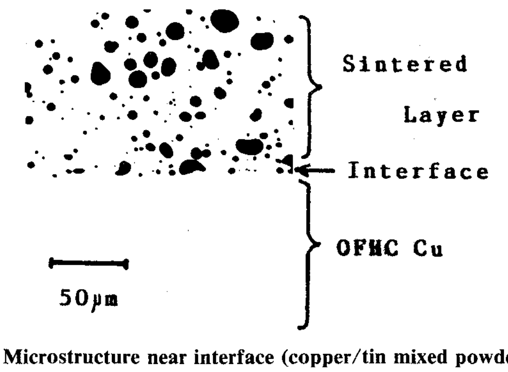


Fig. 6 Microstructure near interface (copper/tin mixed powder and OFHC copper).

On the basis of these experimental data, the optimum conditions for forming the compliant, sintered alloy closeout around the OFHC copper liner of the thrust chamber are: 1) using copper powder with a 10% tin addition, 2) CIP pressure of either 150 or 200 MPa, and 3) sintering at 750°C and then 850°C, each for 1 h in a hydrogen gas flow.

These results were superior to previous results. The tin powder was a concern, because it was thought not to be uniformly mixed with the copper powder in the case of a large structure, such as a trial chamber. It was also expected that more fundamental tests would be needed. Thus, the combination of copper powder and tin plating was selected to fabricate the first trial combustion chamber which demonstrates the desirability of the method.

Fabrication of a Subsized Combustion Chamber

The cylindrical chamber model, as illustrated in Fig. 10, was then fabricated before actually fabricating a subsized combustion chamber. Two types of cylindrical chambers fabricated under the same conditions as mentioned previously were subjected to rupture tests. Model A, which has the same dimensions as the throat section of the trial chamber, was operated at a channel pressure of 100 MPa for 10 min. This indicates that the union has excellent pressure tightness for practical use. Model B, which has a narrower width of ribs than model

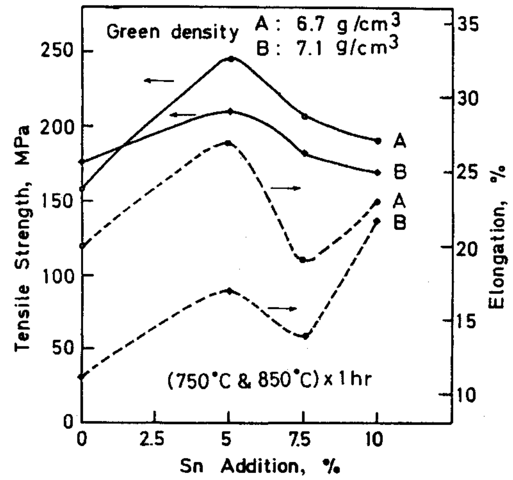


Fig. 7 Tensile strength and elongation of sintered layer.

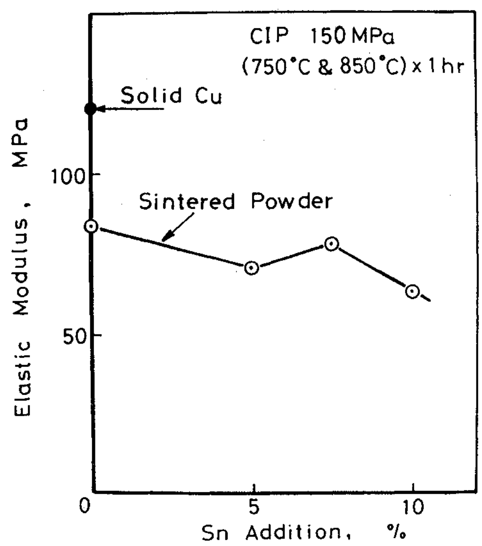


Fig. 8 Effect of tin addition on elastic modulus.

A, was ruptured at a channel pressure of 55 MPa. This indicates, by a simple stress calculation, that the bonding strength between the OFHC copper and the closeout is 165 MPa,

$$S_b = P_j\,(w/r) \tag{1}$$

and obviously more than two times the blanking force ($S_b = 60$ MPa) marked as design point (d.p.) in Fig. 3.

A trial combustion chamber was designed for a chamber pressure of 15 MPa and a thrust level of 10 kN, as shown in Fig. 11. For ease of fabrication, the chamber nozzle was covered with only a sintered copper closeout. The chamber was then fabricated by the CIP forming method under the same conditions mentioned previously, except for the sintering time. The time was changed from 1 h to 100 min because of scale effect.

The fabrication process was as follows: First, the design and fabrication of a wet bag tooling system, suitable for making this combustion chamber, was performed before the cold isostatic pressing step. The chamber was made of OFHC copper and had 36 externally milled slots for the coolant channels. It was then dipped into a melted Woods metal bath. After solidification, the excess Woods metal was removed by machining. This step was performed carefully to prevent casting defects such as shrinkage, cavities, or voids. The design of the wet bag tooling must also be suitable for easy removal of the Woods metal.

The coolant channels filled with Woods metal were then sealed off by electroplating a 10-μm-thick layer of tin over the chamber. Then, a liner and a solid core made of stainless steel were placed in a cylindrical flexible mold. The space between the liner and the mold was filled evenly with copper powder. The powder was isostatically compacted at 150 MPa. The density of the compact obtained was approximately 70% of that of pure solid copper. After cold isostatic pressing, the Woods metal was removed completely by heating the compact at 150°C for 30 min in a protective atmosphere of argon gas. Then the compact was sintered at 850°C for 100 min in a hydrogen gas flow. The sintered density of the closeout was approximately 94% of the theoretical value.

Finally, a sintered closeout was machined into the given size and shape, and then a structural jacket and manifolds were installed in place. The structural jacket was made of stainless steel and designed within the yield strength criterion in order to accept the chamber pressure and external loads. Fusion welding using an electron beam was used to attach the sintering layer and structural jacket at the injector and nozzle ends.

Firing-Test Results

Prior to the firing test, the chamber was hydrotested to assure structural integrity. The pressures during the hydrosatic test were 40 MPa in the coolant channels and 20 MPa in the combustion chamber. A test using liquid hydrogen to confirm durability at an extremely low temperature was also conducted at a pressure of 15 MPa.

Liquid oxygen and ambient temperature gaseous hydrogen were used as propellants and liquid hydrogen was independently supplied as a coolant. The spent hydrogen coolant was disposed of through a vent stack. The firing tests were performed at combustion pressures up to 9 MPa, which was the maximum combustion pressure attainable by the test facility. Total firing time was 300 s for 28 runs.

After the firing tests, the tested chamber was cut. A metallurgical microstructural analysis was undertaken to improve the subsequent fabrication process and conditions. Figure 12 shows a cut view of the tested chamber. Any dimensional changes of the chamber, such as coolant-wall deformation and thinning of a ligament, were not observed because of the small number of firing tests.

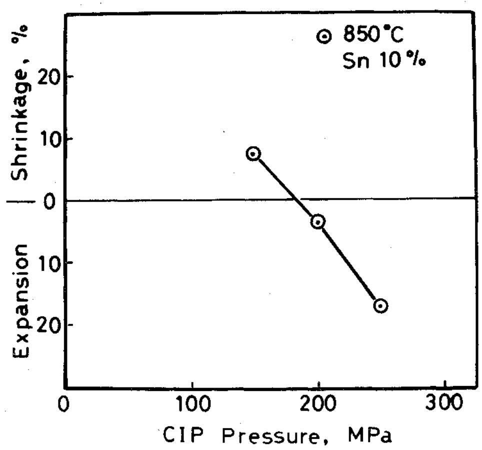
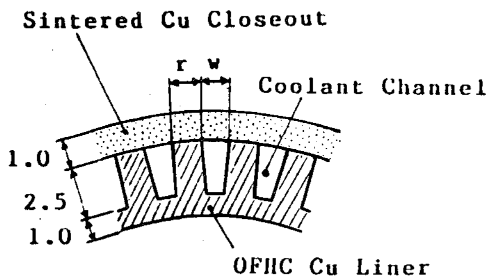


Fig. 9 Dimensional change of sintered body.



Model	(A)	(B) (mm)
w	2.0	3.0
r	2.0	1.0

Fig. 10 Cylindrical chamber model for pressurized tests.

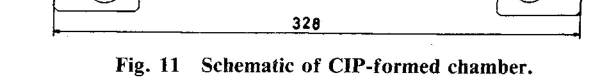


Fig. 11 Schematic of CIP-formed chamber.

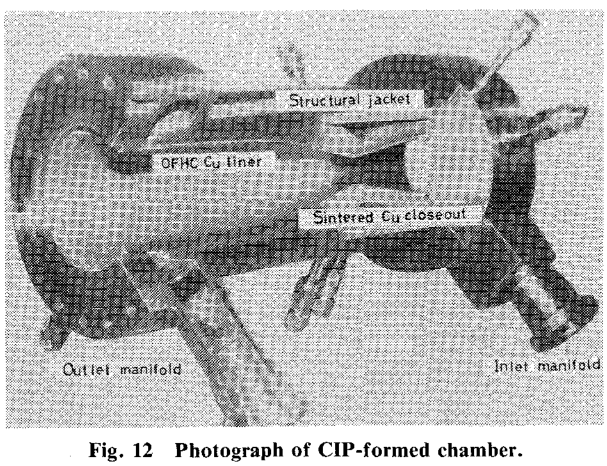


Fig. 12 Photograph of CIP-formed chamber.

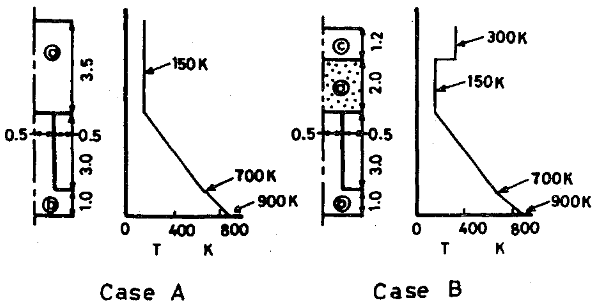


Fig. 13 Structural model: ⓐ, nickel closeout; ⓑ, OFHC copper liner; ⓒ, structural jacket; ⓓ, sintered alloy closeout.

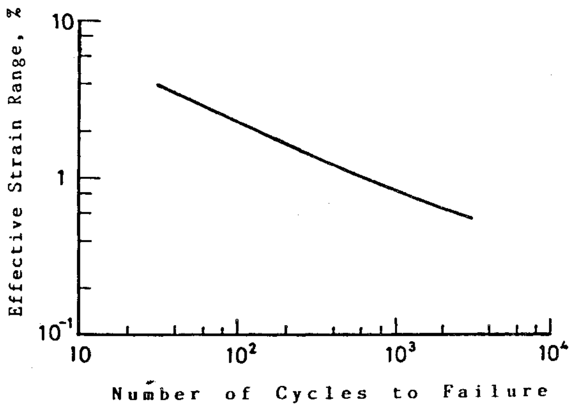


Fig. 14 OFHC copper low-cycle fatigue curve.

Low Stiffness Chamber Structure with Sintered Closeout

Kasper and Notardonato[2] have indicated that thin copper closeouts with low stiffness overwraps of glass fiber can improve the cyclic life of a regeneratively cooled combustion chamber.

This improvement in cyclic life was attributed to the theory that a low stiffness structure would be less constrictive to the thermal growth of the liner during the firing cycle and, thereby, reduces the effective strain range.

A preliminary analysis is shown in this section to demonstrate that a thrust chamber with a compliant, sintered alloy closeout would have a longer life capability than one with a stiffer nickel closeout. Because of symmetry, only one-half of the coolant channel-wall cross sections is shown for two structural models, see Fig. 13. These two models represent sections taken from the throat regions of typical high-pressure thrust chambers fabricated by the conventional nickel electroforming method and the CIP forming method, respectively.

Case A shows a chamber with a stiffer nickel closeout only; where, in practice, a nickel closeout is overwrapped with a stiffer Inconel jacket. For simplicity of modeling, though, an integrated nickel closeout is assumed. Case B shows the chamber with a compliant, sintered alloy closeout overwrapped with an Inconel structural jacket. In case A, the nickel closeout sustains both coolant and chamber pressures. In case B, the sintered closeout contains the coolant pressure, while the external Inconel jacket accepts the chamber pressure. Thermal resistance in the microlayer between the closeout and the structural jacket could easily be assumed to allow the jacket to remain near ambient temperature throughout the firing cycle.

Case A

The average hoop strain range in the ligament, $\Delta\epsilon_p'$, is conservatively evaluated based on the differential thermal expansion of the ligament at maximum temperature T_i, and the closeout at minimum temperature T_0,

$$\Delta\epsilon_{p1}' = \alpha(T_i - T_0) \tag{2}$$

From thermal analysis,

$$T_i - T_0 = 750\ \text{K} \tag{3}$$

Then, for $\alpha = 17.1 \times 10^{-6}\ \text{K}^{-1}$,

$$\Delta\epsilon_{p1}' = 0.0128 \tag{4}$$

On the other hand, as can be seen in Fig. 13, there is a temperature drop through the ligament wall. This also causes plastic straining of the ligament which is constrained at both ends.

According to the theory of Porowski and Badlani,[5] the plastic strain range $\Delta\epsilon_{p1}''$ is

$$\Delta\epsilon_{p1}'' = [E\alpha^2(\Delta T)^2]/[12(1-\upsilon)^2 S_y] \tag{5}$$

The temperature drop across the ligament is $\Delta T = 250$ K. Then, for $E = 117{,}215$ MPa, $S_y = 62$ MPa, and $\upsilon = 0.3$,

$$\Delta\epsilon_{p1}'' = 0.0059 \tag{6}$$

This must be added to the plastic strain range $\Delta\epsilon_{p1}''$ due to thermal expansion to place the plastic strain range into the hoop direction $\Delta\epsilon_{p1}$.

$$\Delta\epsilon_{p1} = \Delta\epsilon_{p1}' + \Delta\epsilon_{p1}'' = 0.0187 \tag{7}$$

Based on the constant-volume assumption of plasticity and the biaxial strain condition, the total effective strain $\Delta\epsilon_{\text{tot}}$ becomes approximately,

$$\Delta\epsilon_{\text{tot}} = 2\Delta\epsilon_{p1} = 0.0375 \tag{8}$$

Case B

It is assumed that thermal expansion in the hoop direction could be absorbed by the compliant, sintered layer. Consequently, only the plastic strain range caused by thermally induced bending remains.

$$\Delta\epsilon_{\text{tot}} = 0.0120 \tag{9}$$

The cycle lives of these two cases were predicted by the low-cycle fatigue curve of annealed OFHC copper, shown in Fig. 14 (Ref. 2) at the calculated strain ranges.

Case A: $Nf = 37$
Case B: $Nf = 390$

The cycle life of the copper liner with a sintered alloy closeout is about 10 times longer than the life of an identical liner with only electroformed nickel under the same conditions of use. The accuracy of this method for life prediction is questionable, but it is believed to be adequate for order-of-magnitude estimates.

Conclusion

1) On the basis of the powder metallurgical preliminary research, the forming conditions for the sintered closeout of the trial combustion chamber were confirmed to be: cold isostatic pressing (CIP) pressure of 150 MPa, tin-plate thickness of 10 μm, and hydrogen sintering at 850°C for 100 min.

2) For the purpose of further improvement of the fatigue property of CIP-formed combustion chambers, the basic metallurgical test was performed, using copper/tin mixed powder instead of copper powder only. The optimum conditions for forming the compliant, sintered alloy closeout were proposed as follows: using copper powder with 10% tin addition, CIP pressure either 150 or 200 MPa, and sintering at 750°C and then 850°C, each for 1 h in hydrogen gas.

3) The trial thrust chamber fabricated by the CIP forming method was test-fired at combustion pressures up to 9 MPa for 28 runs. No dimensional changes were observed after the firing tests and reliability was confirmed.

4) A preliminary analysis also demonstrated that a thrust chamber with a compliant, sintered alloy closeout would have a longer life capability than one with only a stiffer nickel closeout. The cycle life of the OFHC copper liner with a sintered alloy closeout is about 10 times longer than the life of an identical liner under the same conditions, but with only a nickel closeout.

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