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Design of High-Temperature Brazing Alloys for Ceramic-Metal Joints

New alloys show promise of improved joint strength for ceramic heat engine applications

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ABSTRACT. New brazing alloys based on the Au-Ni-Cr-Fe system were developed and tested at room temperature and 650°C for ceramic-metal brazed joints in ceramic heat engines. Mechanical tests included torsion, torsion fatigue, and torsion creep tests with a cylindrical joint geometry. The average torsion joint strength with the new braze was 43.1 N-m at room temperature and 36.0 N-m at 650°C. The rupture life of the joint was within its design goal and exceeded 160 h without a failure at 20.9 N-m (185 in.-lb) torque level. Also, the joints survived over 10^6 cycles for room-temperature fatigue at the torsion amplitude of 3.9 to 20.9 N-m. Improved joint strength at high temperature was attributed to a dual-phase microstructure of the new braze alloys which were strengthened by solid-solution formers such as Cr, Mo and Fe.

Introduction

Joining of ceramic to metal has emerged as one of the critical technologies for the success of ceramic heat engine applications. In recent years, brazing has received renewed attention due to its flexibility in stress accommodation and joint production. Earlier work (Refs. 1–4) focused on identifying material systems for brazing and demonstrating analytical tools for use in designing ceramic-metal joints. The study on the material systems involved the development of a coating layer, interlayer and brazing alloy.

The stress response of joints was investigated analytically as a function of mechanical and physical properties of the joint components. Various constitutive equations were derived for the joint materials as a function of temperature. Several joint geometries and interlayer materials were screened using these equations in finite element analysis (FEA).

Practical problems encountered in joining two dissimilar materials were not only the thermal mismatch, which causes a significant residual stress at the interface, but the chemical compatibility among the joint components and its performance at high temperature. The effort described above resulted in a ceramic-metal joint system that performed well up to 400°C (752°F) (Refs. 3, 4). However, these joints did not provide an adequate torsion strength at 650°C (1202°F), a projected use-temperature of the ceramic-metal joints. This renewed the need for new braze alloys that could meet the high-temperature (650°C) requirements for ceramic-metal joints.

The increase in the use temperature by as much as 250°C required a careful consideration of all joining components and their exposures expected during joining and service operations. The scope of alloy design had to be broadened, but still accommodate a narrow temperature window established between the braze melting point and the upper safe limit of a joining member. The design criteria followed for the new alloys were based on: 1) wettability, 2) melting temperature, 3) room-temperature and high-temperature mechanical properties, and 4) compatibility with other joining components. Figure 1 illustrates the procedure used for the development of high-temperature brazing alloys. Master alloys were selected to meet the requirements of the joints during brazing and applications by studying relevant phase diagrams.

Candidate compositions were evaluated with screening tests, such as sessile-drop wetting tests and hardness tests. The wetting tests were done on coated ceramic and interlayer materials in an argon atmosphere, and Rockwell B hardness was measured. Some promising compo-

KEY WORDS

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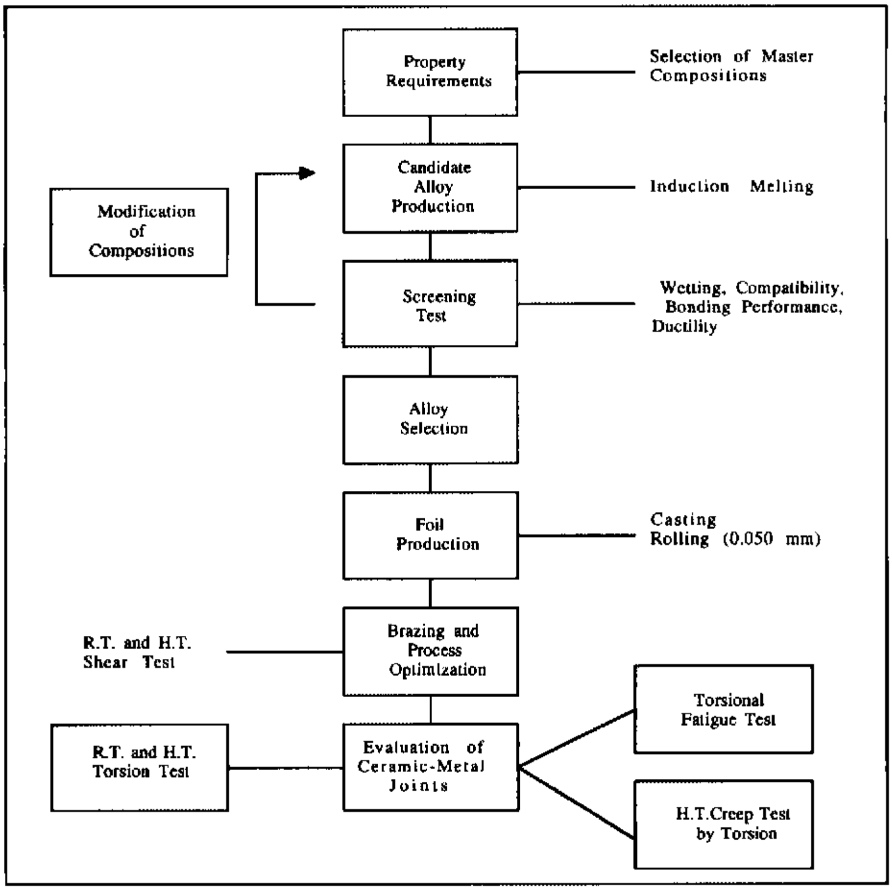

Fig. 1 — Procedure for brazing alloy development. R.T. and H.T. stand for room temperature and high temperature, respectively.

sitions were further modified and refined through a similar screening iteration. This paper presents the results from the mechanical testing of the ceramic-metal joints prepared with new high-temperature brazing alloys, emphasizing the approach for the braze alloy design. The detailed development of new brazing alloys can be found elsewhere (Ref. 5).

Approach for Alloy Design

Finite element analyses performed with various joint materials (Refs. 2, 3) suggested the property requirements for joint component materials. In order to have a reliable performance of the joint at low temperature, the materials should possess such properties as low yield strength, high ductility, and high ultimate strength. High ductility and high toughness of the alloys are regarded as extremely important, not only to accommodate the high residual stresses that develop at the ceramic-metal interface, but to facilitate the rolling of braze foils during fabrication. In addition, the alloys are required to possess an improved creep resistance for high-temperature applications. Many of these characteristics, requisite to the production of the joints and the service requirements, are found to be in conflict with each other. Furthermore, the metallurgical and chemical interactions between joining components, if not taken into design consideration, often cause serious compatibility problems during brazing and use at high temperature.

As the application temperature and time increase, the stability of joining members becomes a critical issue. For example, PY6™ silicon nitride (Si_3N_4 + $6Y_2O_3$, in wt-%) used in this study became unstable, liberating nitrogen gas in the vicinity of 1200°C (2192°F), especially when a reactive coating material was used to promote the wetting of the liquid braze (Ref. 6). Also, an iron-based, low-expansion Incoloy™ 909 (Fe-38Ni-13Co-5Nb-1.5Ti, in wt-%), which is a common choice for this application, is prohibited from any heat treatment above 1200°C. Therefore, the melting temperatures of new brazing alloys were constrained to the range of 1050° to 1150°C (1922°–2102°F) in order to be processed below 1200°C. These additional limitations were reflected in selecting a Ni-based alloy system with controlled amounts of various alloying elements to meet the functional requirements as discussed above.

Many Ni-based alloys display excellent high-temperature strength and toughness. The design strategy was to find alloy compositions which satisfied the requirements above, but still retained room-temperature ductility for the accommodation of residual stress. Solid-solution-strengthened Ni alloys were preferred over precipitation-strengthened ones, which normally contain Ti and Al to form γ' and γ'' in the structure since the latter exhibit a reduced room-temperature ductility. Solid-solution-strengthened Ni alloys, which have Cr, Fe, Mo, and Nb as a strengthener, provide a relatively low yield strength with a high ductility.

Ni-based brazing alloys were designed to maintain a solid-solution, single-phase microstructure without having precipitates after the addition of the alloying element(s). Since most Ni-based alloys have high melting points (>1300°C; 2372°F) and low ductility values relative to the requirements for the joint production, elements such as Au, Cu, Ag (Group IB), Pd or B, Si, Ge or Sn, and In were added to lower the melting points with a minimum loss of ductility. The addition of Group IB elements up to 30% did not reduce the melting points below 1250°C. The addition of various metalloids to Ni alloys to further lower the melting points resulted in a severe embrittlement of the alloy system. Therefore, the scope of the alloy design with Ni-based alloys was altered to focus on Group IB elements (>50%) as base elements to obtain the desired level of ductility.

In order to meet the property goals, the microstructural objective was first established for the system. Relying on known characteristics of the selected system, effort was directed toward developing a composition of a dual-phase, solid-solution microstructure with a stable immiscibility region. The overall design approach for the system is shown in Fig. 2. Among Cu-Ni, Au-Ni, Ag-Ni, and Pd-Ni alloys, which all have immiscibility regions, the Au-Ni system was selected as a base composition. In the course of high-temperature brazing, a decrease in the melting temperature of a braze alloy was often observed due to the influx of other elements from the base materials, i.e., diffusional melting, aborting the production of sound joints. The final base alloy of Au-30 to 50%Ni was selected to take advantage of a stable immiscibility region existing in the system, as well as diffusional solidification. Also, solid solution strengtheners, such as Cr, Mo and Fe, were added to improve high-temperature properties. This approach was confirmed with a typical microstructure of a target composition (Alloy K-1), Au-38Ni-13Cr-5.5Fe-2.5Mo in wt-%, as shown in Fig. 3. This SEM micrograph shows a distinctive dual-phase system: a continuous

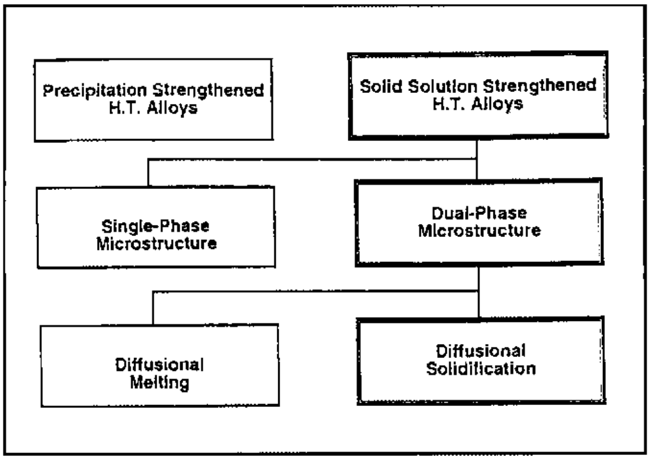

Fig. 2 — Design concept for new braze alloys.

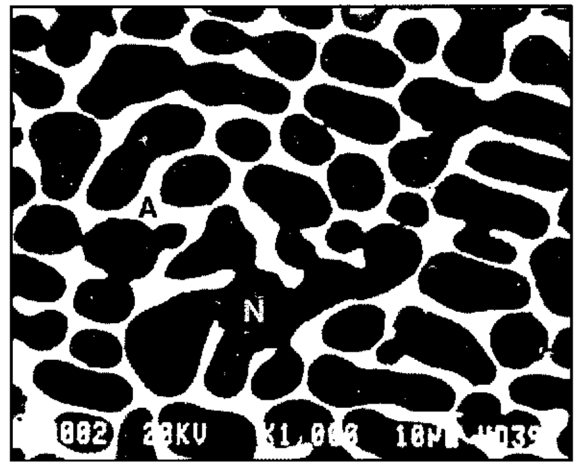

Fig. 3 — Microstructure of K-1 braze. A: Au-rich phase; N: Ni-rich phase.

Au-rich phase with a Ni-rich phase. After extensive screening tests, two alloy compositions, Au-33~35Ni-3~4.5Cr-1~2Fe-1~2Mo (SK-1) and Au-34~36Ni-4~5.5Cr-2~3Fe in wt-% (SK-2), were produced. These alloys showed superior wetting and atomic bonding characteristics, as well as excellent ductility compared to the other compositions studied. The immiscibility region was stable up to 800°C (1472°F) for the selected compositions.

Alloy Production and Analysis

Braze foils were produced for both compositions, SK-1 and SK-2. One hundred twenty grams of each alloy were melted at 1300° to 1400°C (2372°–2552°F) in a boron nitride crucible by induction heating, and cast into a chilled copper mold to produce a 50 x 19 x 6 mm (2 x 0.75 x 0.24 in.) bar. The alloys were homogenized at 800°C for 24 h in an argon atmosphere. The cast bar was rolled into 0.05-mm (0.002-in.) thick foils with intermittent vacuum annealing at 800°C for 2 to 4 h. Rolling of these alloys was successful due to their excellent toughness and ductility.

The brazing foils were subjected to chemical analysis using an ICP (inductively coupled plasma) analyzer. The alloys were homogeneous in compositions without a noticeable deviation from the target compositions. Differential thermal analysis (DTA) was done for SK-1 and SK-2 to characterize their thermal transition behaviors. Both alloys exhibited two discrete melting events. They were related to the existence of two major phases in the microstructure. The one phase melted in the vicinity of 1000°C (1832°F) and the other phase between the temperature range of 1260° to 1270°C (2300°–2318°F). The addition of Cr, Fe and Mo yields two melting points in the new alloys, differentiating them from a Au-Ni binary alloy. Based on these results and the temperature requirement for a desirable ceramic-metal joining process, the brazing temperature range for these alloys was set at 1050° to 1150°C.

For the study on microstructure, PY6/Ni lap joints were prepared using the SK-1 and SK-2 alloys. Nickel was used throughout the experiments as an interlayer material to minimize the residual stress at the ceramic-metal interface (Refs. 4, 5). Also, for ceramic-metal brazing, PY6 was coated with 3-μm-thick Ti to promote the wetting of liquid braze. The coating was done, using an electron beam deposition technique. The general microstructures of the joints are shown in Figs. 4 and 5 for the SK-1 and SK-2 alloys, respectively. The phase distribution was determined with the use of a microprobe. As expected, both alloys exhibited two discrete phases in the structure (Au-rich and Ni-rich) with an irregular phase boundary. The irregularity of the phase boundary and the reduced volume of the Au phase were due to the Ni and Ti diffusion from the Ni interlayer and Ti-coating layer on the ceramic. The microstructure of the brazed joints consistently showed a fine layer of the low-melting Au-rich phase at the braze/ceramic interface. This reveals that the atomic bonding between ceramic and Ni interlayer relies on the spreading of the Au-rich phase. This phase was absent at the braze/Ni interlayer interface.

As shown in Fig. 4, the Au-rich phase in Alloy SK-1 consists of Au and Ni with minimal amounts of Cr, Fe and Mo. Mo shows a nonuniform distribution in the Au-rich phase of SK-1 used for brazing. The result is consistent with a low solid solubility limit of Mo in Au: almost 0% at room temperature and about 1% at 1054°C (1929°F). However, little Mo was found in the Ni-rich phase as contrast to that of K-1 alloy where Mo was always associated with Ni-rich phase. The Ni-rich phase consists mostly of Ni, Cr, and Fe in the form of a solid solution. This phase is essential for creep strength at high temperature. The Ti-coating remained intact at the PY6 surface without complete dissolution in the Au-rich phase. The EDAX results of both alloys showed a similar elemental distribution in the Au-rich and Ni-rich phases.

Performance Tests of Ceramic-Metal Joints

Strength Evaluation of Joints

Shear strengths of various lap joints were measured at room temperature, 500°C (932°F), and 650°C (1202°F) to compare the performance of SK-1 and 2 with those of reference alloys such as Au-5Pd-2Ni (Ref. 4) and Au-18Ni. The joints were made between PY6 and Ni interlayer as for the microstructural analysis. The intended area for joining was 1 cm^2 and the thickness of the braze alloys was 0.050 mm. The results are summarized in Table 1. In general, the new braze alloys outperformed Au-Pd-Ni and Au-Ni at high temperature. However, the performance of SK-2 at room temperature was found to be somewhat low and inconsistent compared to Au-Pd-Ni or Au-Ni. The low room-temperature strength indicates that the braze alloys are less ductile than Au-Pd-Ni or Au-Ni. It is known that at low temperature the joint strength is controlled by the capability of the braze alloy and metal substrate to accommodate the residual stresses. At high temperature, the presence of a multi-phase microstructure and reduced residual stress enhanced the joint strength. Au-Pd-Ni and Au-Ni showed better performance at low temperature, but they exhibited no strength at high temperature.

Torsion Testing

Cylindrical joints were produced for

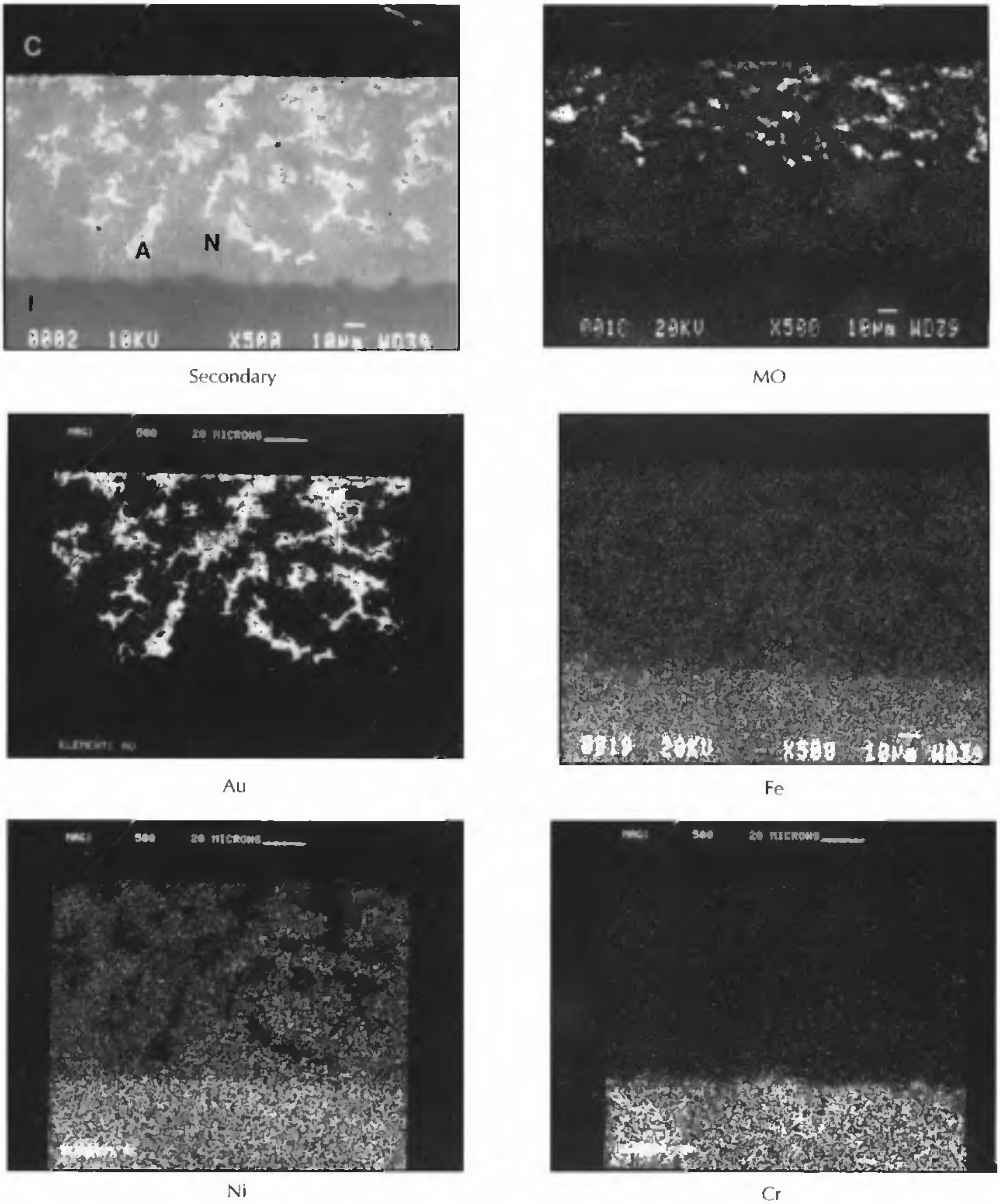

Fig. 4 — Microstruture of SK-1 braze. C: PY6 ceramic; I: Ni interlayer; A: Au-rich phase; N: Ni-rich phase in the braze.

the PY6-Ni-Incoloy 909 system, using the new braze alloys, to evaluate the torsion strength and torsion fatigue strength. A ring-shaped Ni interlayer and Ti coating were used for the reduction of residual stress and the improved wetting, respectively. The cross-sectional view of the cylindrical joint geometry is shown in Fig. 6. PY6 silicon nitride of 12.7-mm (0.5 in.) in diameter and Incoloy 909 were chosen for their excellent high-temperature strength and low thermal expansion coefficient, respectively. The surfaces of PY6 were polished down to 0.03 μm with alumina powder for brazing. Ti was coated on the intended joint area, 2 cm^2, of the ceramic. A detailed description on the joint geometry can be found elsewhere (Ref. 7).

All torsion tests for the brazed joints were performed with an MTS Model 646.25S hydraulic axial/torsion machine. The maximum torque of the machine is 2260 N-m, and the maximum axial load is 244 kN. The machine was equipped with hydraulic grips that accommodated 12.7-mm-diameter samples. The gripping pressure was adjusted to 10 MPa to protect the ceramic from premature cracking in the gripped area

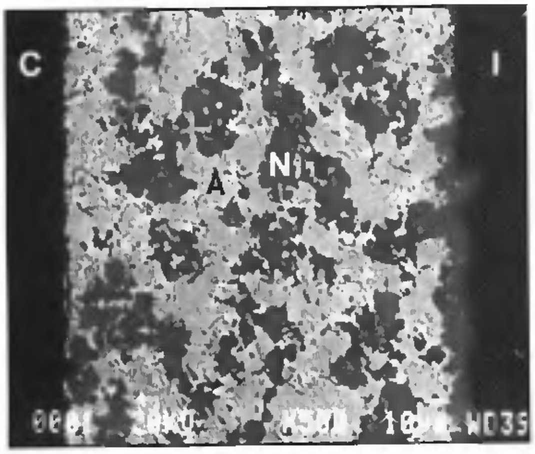

Fig. 5 — Microstructure of SK-2 braze. C: PY6 ceramic; I: Ni interlayer; A: Au-rich phase; N: Ni-rich phase in the braze.

during testing. The torsion tests were run at a rate of 0.5 to 1.7 N-m/s in axial load and torque control modes. The axial load was maintained to within 14.5 N. An induction furnace was used to heat the joints to 650°C for the elevated temperature tests. A SiC susceptor was used to produce uniform heating of the ceramic-metal joint. The joints were held at the test temperature for 30 min before each test. Room-temperature torsion tests were performed with cylindrical samples of 12.7 mm diameter x 100 mm (4 in.) long.

The test results with the brazed joints are shown in Table 2. The brazing alloys, especially SK-1, gave a significant increase in torsion strength at 650°C. A moderate loss in strength was observed at room temperature compared to earlier work done with the Au-5Pd-2Ni braze alloy (Ref. 4). The average joint strength made with SK-1 was 36 N-m at 650°C, whereas the strength at room temperature was 43 N-m. The 650°C torsion strength was far better than what we observed from the Au-5Pd-2Ni system, 1.1-6.8 N-m. The SK-1 alloy seemed to maintain the room temperature strength well up to 650°C with little scatter in the failure strength values. The SK-2 alloy also exhibited a moderate gain in strength at high temperature compared to that of the Au-5Pd-2Ni system. The results from SK-2 scattered greatly due to the inconsistency in the joint production.

It was noted that the torsion failure at 650°C occurred in the PY6 ceramic when SK-1 or SK-2 was used. It means that the atomic bond strength between the braze alloys and silicon nitride exceeds the strength level of the ceramic at 650°C. The joint failures in the previous study were always associated with the failure of brazing alloys. Therefore, the average torsion strength of PY6 silicon nitride itself in this study was measured. They were 238 and 59.3 N-m at room temperature and 650°C, respectively, which are about 85% of the values found in the previous study (Ref. 5). This indicates that the performance of the joints with new brazes could be better than what were observed in this study. The low strengths of these joints at room temperature, compared to the Au-5Pd-2Ni system, were obtained at the expense of the improved high-temperature strength of the SK-1 and SK-2 alloys.

Torsion Creep Testing

It was found (Ref. 2) that the joints made with Au-5Pd-2Ni could not sustain any significant load (>2.3 N-m) at 650°C, even though they performed well at room temperature. Table 3 summarizes the creep test result of SK-1 joints at 650°C. At 20.9 N-m (185 in.-lb) torque level, the rupture lives of four joints were 3.5 h, 10.5 h, >100 h, and >160 h with creep rates of 4.5×10^{-2}, 6.7×10^{-2}, 8.8×10^{-4}, and 4.6×10^{-3} deg/h, respectively. Failure of the first two joints in the table occurred by rupture failure at the interface between the braze and PY6. These samples showed a relatively high unbonded area (>30%) in the joint. Testing of the other two joints were stopped after 100 h and 160 h of creep without a rupture. The performance of these two joints at 650°C was exceptionally good.

The first sample had a rotational creep rate of 0.045 deg/h, and failed when it rotated about 0.22 deg beyond the end point of first stage creep (0.50 deg). The steady-state creep of the third sample initiated at 0.552 deg and stopped at 0.640 deg after 100 h, resulting in a creep rate of 0.00088 deg/h. A significant improvement in the creep rate of the third sample was most likely related to the properties of the braze alloy and to the quality of the joined area, *i.e.*, the percentage of unbonded area and its distribution. Figure 7

Table 1 — Results of Shear Tests at Various Temperatures

System	Brazing Alloy	Avg. RT	Strength[a] 500°C	(ksi) 650°C
PY6[b]-Ni	Au-Pd-Ni (ref.)	—	8.5	6.8
	Nioro(ref.)	8.2	13.5	12.3
	SK-1	8.6	14.2	14.5
	Sk-2	5.4	15.2	11.3

(a) Strength values are avg. of min. two tests.
(b) PY6 was coated with 3 μm Ti for wetting of brazing alloys.

Table 2 — Torsion Strength of the PY6/Ni/Incoloy 909 System Brazed with SK-1 or SK-2 Braze Alloy at 1100–1150°C for 30 Minutes

Braze Alloy	Test Temperature (°C)	Torque (N-m)	Alignment (microns)	Rotation (deg)	Failure Mode
Au-Pd-Ni (4)	RT	Avg. 61	—	—	Ceramic fracture
	650	Avg. 4.5	—	—	Slipped
SK-1	RT	45.8	75	1.23	Ceramic fracture
		47.5	200	1.30	Ceramic fracture
		34.4	125	0.88	Ceramic fracture
		35.0	150	—	
		44.1	150	—	
		45.2	350	1.15	Ceramic
		49.7	250	1.25	Ceramic
	650	34.7	375	1.23	Ceramic fracture
		32.8	275	0.95	Ceramic fracture
		37.3	475	1.20	Ceramic fracture
		16.4[a]	350	—	>50% unbonded
		38.4	250	1.20	Slipped
		8.1[a]	375	0.37	Slipped
SK-2	RT	45.2	500	1.25	Ceramic fracture
		45.2	—	—	Ceramic fracture
		35.6	125	0.92	Ceramic fracture
		39.5	100	0.96	Ceramic fracture
		27.1	50	—	Ceramic fracture
		53.7	—	—	
		73.4	350	2.25	
		72.3	375	1.87	
	650	36.7	225	—	Ceramic fracture
		1.7	125	—	>50% unbonded
		10.2	—	—	Slipped
		1.1	450	—	
		8.5	700	0.27	
		5.6	—	0.15	

(a) These values were regarded as outliars and ignored for the calculation of the average strength.

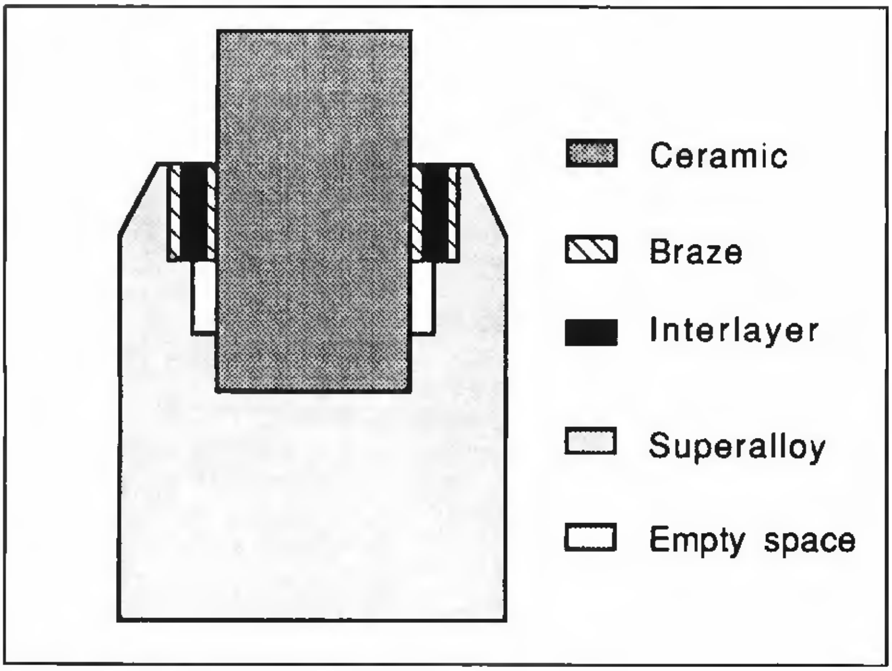

Fig. 6 — Schematic of the cylindrical joint geometry.

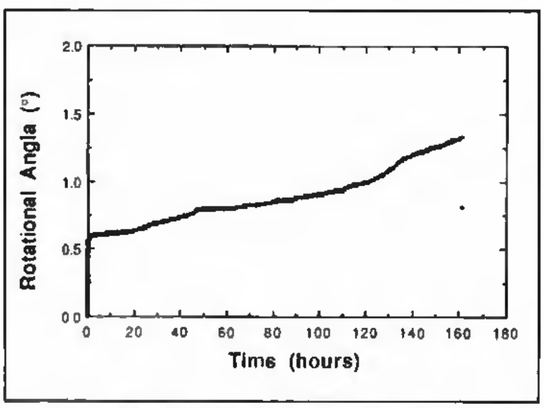

Fig. 7 — Creep behavior of ceramic-metal joints brazed with the SK-1 alloy at 650°C.

Table 3 — Torsion Rupture of PY6/Ni/Incoloy 909 Joints at 650°C

Braze Alloy	Constant Torque (n-m)	Alignment (microns)	Rotation (deg)	Time to Rupture (h)	Creep Rate (°/h)
SK-1	20.9	850	0.58	3.5	4.5×10^{-2}
	20.9	550	1.265	10.5	6.7×10^{-2}
	20.9	625	0.640	>100[a]	8.8×10^{-4}
	20.9	200	1.317	>160 (1 week)	4.6×10^{-3}

(a) Experiment was stopped after 100 hours creep at 650°C.

Table 4 — Torsion Fatigue of the PY6/Ni/Incoloy 909 System

Braze Alloy	Test Temperature (°C)	Torque Amplitude (N-m)	Alignment (microns)	Rotation (deg)	Number of Cycles for Failure
Au-5Pd-2Ni	RT	3.95–20.9	—	—	>1,000,000
(Refs. 1,4)		7.9–41.8	—	—	>1,000,000
SK-1	RT	3.95–20.9	400	0.59	427,440
		3.95–20.9	500	0.51	>1,000,000
	650	3.95–20.9	625	—	Loading Failure

shows a plot of creep behavior of the fourth sample, which survived more than 160 h (one week) at 650°C. The average creep rate of the second stage was 0.0046 deg/h, which is five times higher than that of the third sample. Assuming that the third sample creeps to 1.32 deg of rotation as the fourth sample did, the useful life of the third sample could be more than 850 h. This creep life is quite encouraging for the future applications of these braze alloys.

A significant difference in the creep rate was recorded during the second stage of creep. The changes in the creep rate always coincided with temperature fluctuations between 620° and 670°C. This implies that a small reduction in use-temperature (*e.g.*, 600°C) could improve the rupture life of the joint remarkably. The heating for the torsion creep tests was also done in air, using an induction heating unit. As shown in Table 3, the creep rate is not related directly to the rupture life of the joint. During the high-temperature creep, most deformations were expected to occur in the Au-rich phase, while the Ni-rich phase provided a resistance to the flow of the Au-rich phase. However, the existence of two phases of different melting points necessitates the development of an optimum processing cycle for brazing to ensure the reliability of the joint.

Fatigue Testing

The thermal fatigue resistance of the cylindrical joints (PY6-Ni-Incoloy 909) was evaluated by subjecting them to 1000 thermal cycles between 335° and 650°C with a time interval of 130 s. Thermally fatigued joints were then examined in microfocus x-ray radiography for the development of cracks in the ceramic part. It was found that none of the joint showed any evidence of cracking within the limit of x-ray detectability of about 1 µm.

The mechanical fatigue tests were also done under torque control along with axial load controlled to within ±4.4 N throughout the testing. The tests were conducted between 3.9 and 20.9 N-m (35–185 in.-lbs), with a mean torque level of 12.4 N-m, which represents conditions expected in an automotive engine during idling and at maximum speed (rpm). A loading frequency in the range of 0.016 to 1.5 Hz was used for room-temperature fatigue testing.

The results of two joints tested at room temperature are listed in Table 4. All of the tested joints easily met the service requirements and any further testing was deemed unnecessary. The room temperature fatigue property of the 5K-1 joint was found to be excellent, far exceeding the specified number of cycles (1000 cycles) at the torque amplitude. No 5K-2 joint was tested in fatigue. For high-temperature fatigue testing, the parameters such as torque loading rate, *i.e.*, 10 to 15 s to reach full speed, and duration time at maximum and minimum torque values, 10 to 20 s, and loading frequency, 0.016 Hz, were determined based on the expected performance of an automotive engine. Only two mechanical fatigue tests were performed at 650°C, and both failed in the vicinity of 15.3 N-m toque during loading. Based on the previous results and the limitation in the number of tests, this premature failure was ascribed to the quality of the joints.

Summary and Conclusions

Two alloy systems, Au-Ni-Cr-Fe-Mo (SK-1) and Au-Ni-Cr-Fe (SK-2), were developed to satisfy the requirements of high-temperature performance at 650°C for ceramic-metal joints in ceramic heat engine applications. In the design of new alloys, the microstructural criterion of both solid-solution and dual-phase strengthening was employed for high-temperature brazing alloys. Also, an effort was made to balance the property requirements of the braze alloys for the ease of manufacture and high-temperature properties.

The new alloys provided superior joint performance at high temperature, as compared to conventional solid-solution braze alloys. The SK-1 alloy resulted in a significant gain in the torsion strength at 650°C for PY6-Ni-Incoloy 909 joints with a moderate loss at room temperature compared with baseline Au-Pd-Ni joints. The average SK-1 joint strength was 35.8 N-m at 650°C, whereas the strength at room temperature was 43.1 N-m. The high-temperature torsion strength was far better than the Au-5Pd-2Ni braze alloy system (1.6–7.7 N-m).

The creep performance of these joints at 650°C was outstanding. The rupture life of the SK-1 braze joint exceeded 160 h at the 20.9 N-m (185 in.lb) torque level. Most rupture failures occurred at the interface between braze and PY6. Excellent performance was obtained when the atomic bonded area exceeded 80%. In addition, excellent room-temperature mechanical fatigue property, as well as thermal fatigue resistance, were noted for the SK-1 joint at a fatigue amplitude of 3.9 to 20.9 N-m. The significant improvement in high-temperature torsion strength and creep performance is ascribed to the dual phase microstructure of the SK-1 alloy. The microstructures of the joints made of the alloys show two discrete Au- and Ni-rich phases, which have different melting points.

Acknowledgments

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