

BRAZING AND DIFFUSION BONDING OF GLIDCOP[®]

DISPERSION STRENGTHENED COPPER

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Abstract

GlidCOP[®] Dispersion Strengthened Copper ("DSC") consists of a pure copper matrix with a small amount of (0.7 to 2.7 vol. %) sub-microscopic Al₂O₃ particles. The material is fine grained and it resists recrystallization at elevated temperatures, including those used for brazing with silver and gold base braze alloys. Problems are encountered in the brazing of GlidCOP with silver base braze alloys, due to excessive diffusion of silver along the grain boundaries. Electroplating with copper or nickel, prior to brazing, solves these problems. Various joining and brazing techniques that are effective in producing sound braze joints are discussed here. Excellent braze joint strengths are also achieved with Au-Cu braze alloys. GlidCOP[®] DSC can also be joined to itself or stainless steel via diffusion bonding. Joint strengths of various brazed and diffusion bonded samples are presented.

Introduction

A family of dispersion strengthened coppers, commercially known as GlidCOP[®], possesses tensile strengths ranging from 380 MPa to 700 MPa, with electrical conductivities in the range of 78 to 92% IACS. These materials comprise of a pure copper matrix which is strengthened by a uniform dispersion of 3 to 12 nm. size Al₂O₃ dispersoids^{1,2}. The dispersoids are stable at elevated temperatures, up to the melting point of the matrix, and prevent recrystallization and softening of the material when exposed to high temperatures. Table 1 lists the room temperature mechanical and physical properties of the three grades of GlidCOP[®]. These combinations of mechanical strength and electrical (and thermal) conductivity are rarely found in other copper base materials.

The softening resistance of GlidCOP, as a function of the annealing temperature, is shown in Figure 1, along with those of OF copper and zirconium copper. As shown here, the change in the yield strength of GlidCOP due to high temperature exposure is minimal compared to those of OF copper and zirconium copper. Other precipitation strengthened copper alloys also undergo rapid softening, as a result of coarsening

and dissolution of the precipitates in the matrix, at or below the temperatures normally used for brazing. Since the dispersoids in GlidCOP are inert and do not change in size and distribution, the electrical and thermal conductivities of GlidCOP remain unaffected by exposure to high temperatures. For these reasons, GlidCOP fits well into applications where high temperature joining techniques are used.

Table 1: Room Temperature Properties of GlidCOP[®]

Property		Grade: AL-15	Grade: AL-25	Grade: AL-60
Al ₂ O ₃ Content	Weight %	0.28	0.46	1.12
Electrical Conductivity,	% IACS	92	87	78
Thermal Conductivity,	W/(m k)	365	344	322
Yield Strength, MPa	As Cons.* 75% C.W.	331 455	345 467	413 572
Tensile Strength, MPa	As Cons. 75% C.W.	413 483	434 524	517 627
Elongation Percent	As Cons. 75% C.W.	24 10	21 9	13 9

(*As Cons. - As Consolidated)

GlidCOP is not, however, amenable to joining by fusion welding, including electron beam welding. Remelting of the copper matrix leads to agglomeration of Al₂O₃ dispersoids and recrystallization of the copper matrix, in the weld zone. Most of the agglomerated Al₂O₃ may slag off, while some may remain trapped in the re-solidified metal as inclusions causing the weld zone to be brittle. Minimization of the volume of base material remelted, as is often possible in electron beam welding, can reduce the amount of agglomerated Al₂O₃ and, therefore, reduce the negative effects of this method of joining.

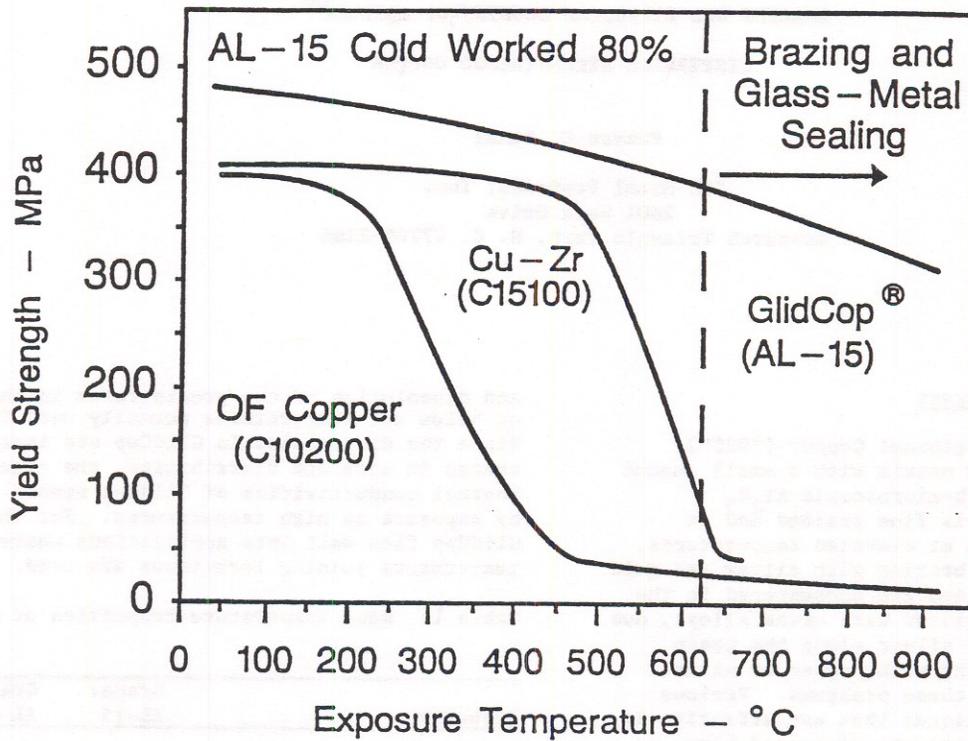


Figure 1: Softening resistance of GlidCop AL-15 versus OF copper and Zirconium Copper. (Note: Properties measured at room temperature, after exposure to elevated temperatures for one hour.)

Brazing with Silver Base Braze Alloys

Since GlidCop is comprised of a pure copper matrix, with less than 3 volume percent aluminum oxide, most brazing filler metals that are commonly used for joining plain copper can be used for brazing GlidCop. However, when brazing with silver-base braze alloys, certain problems are encountered due to the fine grain structure of GlidCop. Copper and most copper alloys undergo recrystallization and grain growth during brazing thus resulting in a coarse grained base material. GlidCop has a fine grain structure and it does not recrystallize during brazing. The diffusion rate of silver along the grain boundaries is much greater than it is through the volume of grains. The large network of grain boundaries in GlidCop, since it is fine grained, permits rapid migration of silver into the material. The rate of migration is so high that in most furnace brazing operations it leads to substantial depletion of silver in the braze joint.

Excessive diffusion of silver along the grain boundaries is often accompanied by the formation of small voids in the material, near the braze joint. Figure 2 is a scanning electron micrograph, in the back-scattered mode, showing the cross-section of a GlidCop brazement made with the Ag-Cu eutectic alloy (BAg-8). The GlidCop samples were not plated and the braze cycle was 15 minutes long. In the back-scattered mode, the silver rich areas appear light in

color. As may be seen in this figure, voids have developed in GlidCop along with the grain boundary migration of silver. This, of course, can lead to embrittlement of the joint.

Electroplating of the surfaces of the parts with either nickel or copper prior to brazing is an effective means of overcoming the problem of silver diffusion. The plated layer acts as a barrier to diffusion of silver. Figures 3(a) thru 3(d) show schematically the Ag diffusion profiles for GlidCop, with and without diffusion barriers, and also that for plain copper. As may be seen in Figure 3(a), for unplated GlidCop, the net migration of Ag (area under the curve to the right of braze alloy interface) is extremely high. In the case of unplated plain copper, Figure 3(b), silver diffusion also takes place mainly along the grain boundaries, but since it has very few grain boundaries (per unit volume) the net migration of silver is minimal. When GlidCop is plated with nickel, the silver concentration across the plated layer is extremely small (less than 1%), as shown in Figure 3(c). The silver concentration in the nickel layer drops rapidly due to the low rate of diffusion of silver in nickel. Figure 3(d) shows the Ag diffusion profile for a copper plated sample. The depth of penetration of Ag in this case will depend upon the grain structure and uniformity of the thickness of the plated deposit.

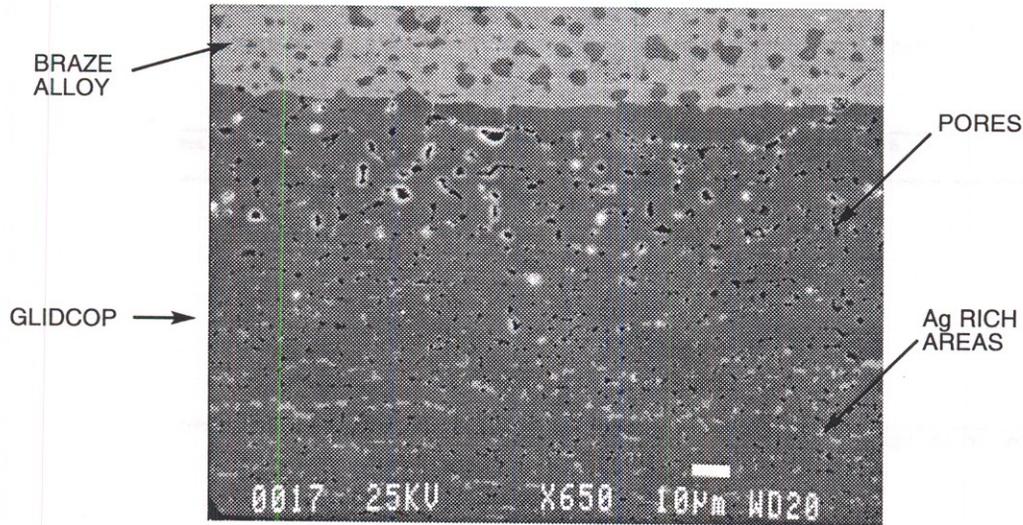


Figure 2: Scanning Electron Micrograph of an unplated GlidCop brazement showing undesirable diffusion of silver along grain boundaries and the associated porosity.

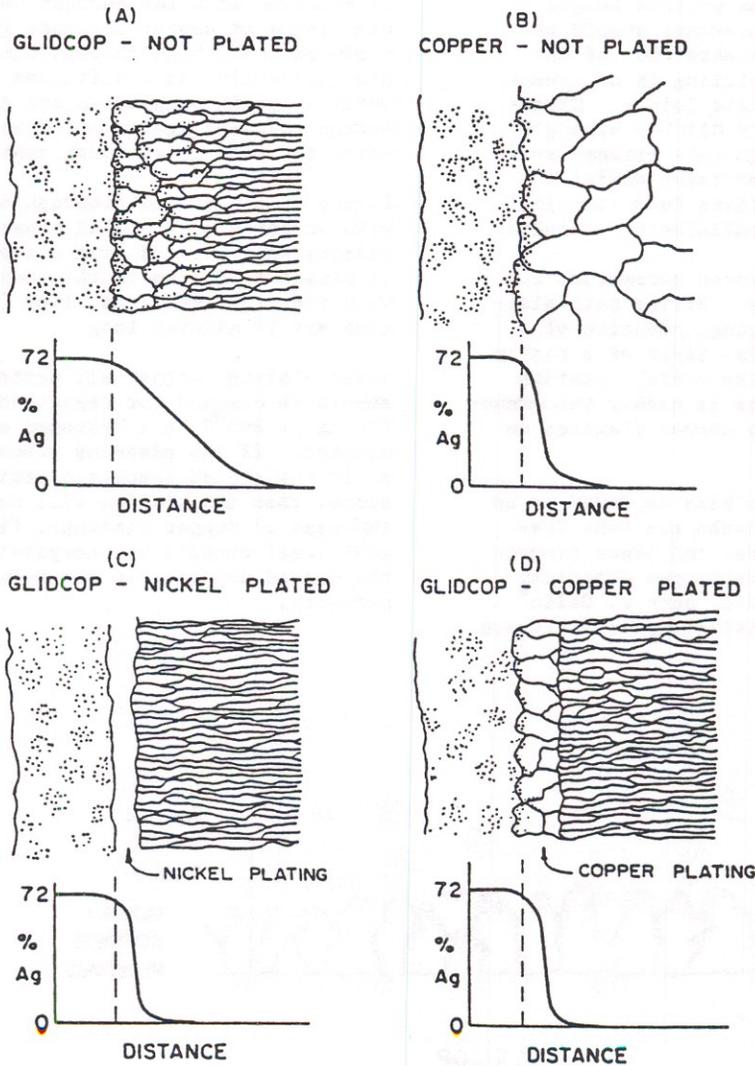


Figure 3(a) - 3(d): Schematic sketches showing diffusion profiles of silver near the braze joint for GlidCop, with and without plating (diffusion barrier) and for plain copper.

Table 2: Recommended Plating Thickness

Brazing Temperature	Brazing Time** in Minutes	Minimum Plating Thickness, mm	
		Copper	Nickel
746° C	5	.015	.005
	10	.020	.008
815° C	5	.020	.008
	10	.030	.012
871° C	5	.030	.012
	10	.040	.015

(* Time period at or above solidus temperature)

Electroplating

The minimum required plating thickness for a given brazing operation is a function of the braze temperature and the braze time. Table 2 lists recommended plating thicknesses for various braze temperatures and braze time periods. For time periods longer than those shown, the plating thickness should be increased in proportion to the square-root of the time period. Adherence of the plating is of utmost importance to achieving sound braze joints. Cleanliness of the surface of machined GlidCop strongly influences adherence. An inadequately cleaned surface leads to blistering of the plated layer during the brazing operation. Appendix I lists four cleaning procedures, all of which yield satisfactory results.

Appendix II outlines the recommended parameters for the strike and the plating baths. Strike bath platings, which precede regular plating, comprise of a very thin (2 to 3 microns) initial layer of a highly adherent deposit that enhances the overall plating adherence. A copper strike plate is highly recommended for nickel platings. Cyanide copper platings do not require a pre-plate strike.

For nickel plating, a dull Watts bath is found to be the most reliable. Dull Watts baths are kept free of brighteners and other additives, and hence produce a pure nickel deposit, free of entrapped organics. Tankless electroplating techniques, such as Dalic selective plating, are also satisfactory as pre-braze nickel platings.

For copper plating, cyanide bath platings are found to be most satisfactory. An acid copper sulfate bath can also produce a satisfactory deposit provided that the bath is kept free of organic additives and is operated at a low current density. Deposits consisting of coarse columnar grains, such as those produced under high current density conditions, are ineffective as a diffusion barrier, because the vertical grain boundaries and gaps formed between the coarse columnar grains offer undesirable diffusion paths for silver, as shown schematically in figure 4.

Figure 5 is a photomicrograph of a braze joint made with an acceptably smooth copper plating. The plating was obtained from a copper cyanide bath and it measured .015 mm in thickness. Brazing was made with the Ag-Cu eutectic alloy (BAg-8) and the braze time was 15 minutes long.

After plating, either all parts or a few samples, should be checked for resistance to blistering by firing at 800° C in a hydrogen atmosphere, for 5 minutes. If the cleaning procedure is inadequate or if the plated deposit contains any organic inclusions, then the plating will develop blisters. In the case of copper platings, firing may provide some additional benefit by recrystallizing and densifying the plated deposit, in the event it contains some porosity.

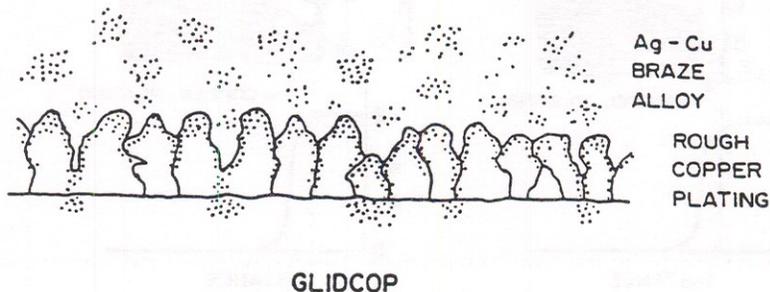
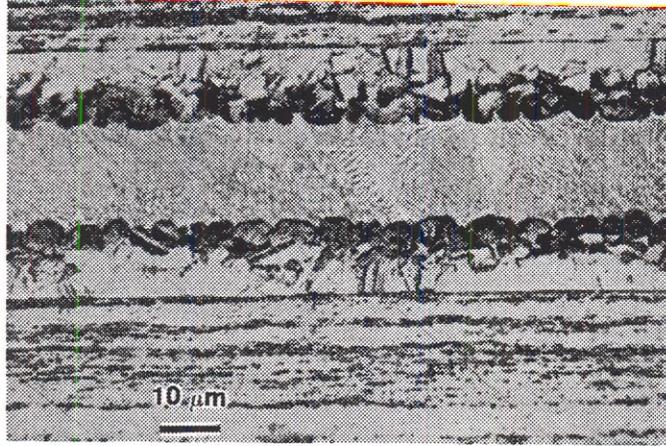


Figure 4: Sketch showing undesirable diffusion of silver along grain boundaries and gaps between the coarse columnar grains of a rough copper plating.



COPPER
PLATING

BRAZE
ALLOY

COPPER
PLATING

GLIDCOP

Figure 5: Photomicrograph of a GlidCop - GlidCop brazement made with acceptably smooth copper plating. Copper plating was obtained from a copper cyanide bath.

Braze Joint Strengths

In order to determine the effectiveness of various plating materials and thicknesses, samples of GlidCop AL-15 and AL-25 were brazed under various conditions and were tested in shear. The sample configuration and test procedure were based on the American Welding Society's "Standard Method for Measuring the Strength of Brazed Joints".

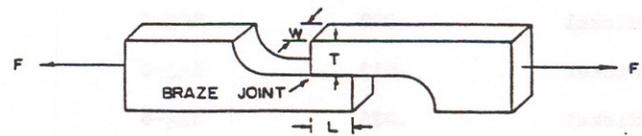
Figure 6 (a) shows the specimen geometry and the test configuration. For each combination of plating and brazing parameters, at least two sets of samples were brazed with varying values of L/T (overlap/thickness). Each assembly was held in a fixture to ensure proper alignment, the joint gap and the desired overlap distance. The braze joint gap was kept at 0.15 mm for all tests. The shear test data for each set of plating and brazing parameters was plotted on a log-log plot of shear stress at failure vs. L/T. From this plot, the shear strength value for L/T = 1 was obtained by interpolation. A typical plot of shear strength vs L/T is shown in Figure 6(b). Table 3 lists the shear strength data for AL-15. Also listed in this table, for comparison, are some data from similar work done at Varian and TRW.

In addition to the four braze alloys listed in this table, tests also included samples brazed with several other silver base braze alloys, namely; BAg-Cu-24%, Ag-14.5%, Cu-28.5%, Ni-2.5%, Pd-10%, Palcusil 10 (Ag-58.5%, Cu-32%, Pd-10%) and Braze 630 (Ag-63%, Cu-28.5%, Ni-2.5%, Sn-60%). All these alloys were found to produce satisfactory results when used in conjunction with a nickel or copper plating. A number of tests were also performed on AL-25 specimens and the results were very similar to those of AL-15.

For the purpose of evaluation, a joint strength of 173 MPa (25,000 PSI) or higher was considered acceptable and a joint strength of 173 MPa (25,000 PSI) or higher was considered excellent, since the latter is the typical shear strength of most silver base braze alloys.

An examination of the data in Table 3 shows the following:

(a) In the torch brazing of GlidCop, satisfactory joint strengths can be obtained without any plating. Metallographic examination of these samples showed that although some silver diffusion had taken place, no void formation had occurred due to the short time periods involved.



SHEAR STRESS = $F \div (L \times W)$

Figure 6(a): Shear Test Arrangement

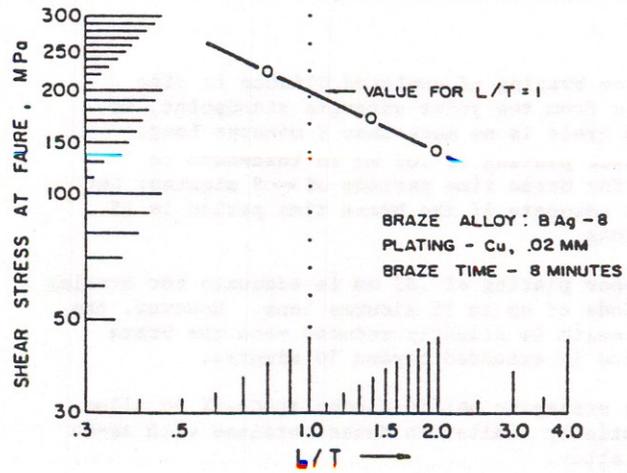


Figure 6(b): A typical plot of shear strength versus overlap to thickness ratio (L/T)

Table 3: Shear Strengths of GlidCop AL-15 Brazements made with Silver Base Braze Alloys (L/T=1.0)

Plating Type	Thickness mm	Braze Alloy	Braze Time minutes	Shear Strength MPa	Data Source
None	--	BAG-1	Torch Braze	166	SCM
None	--	BAG-2	Torch Braze	169	SCM
None	--	BCup-5	Torch Braze	167	SCM
None	--	BAG-8	2	159	SCM
None	--	BAG-8	2	136	Varian
Copper	.010	BAG-8	2	207	Varian
Copper	.020	BAG-8	8	206	SCM
Copper	.020	BAG-8	15	128	SCM
Copper	.030	BAG-8	3	203	SCM
Copper	.030	BAG-8	5	200	SCM
Copper	.030	BAG-8	10	200	SCM
Copper	.030	BAG-8	15	179	SCM
Copper	.015	Ag-Cu-5% Pd	5	228	SCM
Copper	.020	Ag-Cu-5% Pd	10	187	SCM
Nickel	.008	BAG-8	5	178	SCM
Nickel	.008	BAG-8	10	162	SCM
Nickel	.013	BAG-8	2	159	Varian
Nickel	.025	BAG-8	5	143	TRW
Nickel, w/copper strike	.015	BAG-8	10	202	SCM
Nickel, w/copper strike	.015	BAG-8	15	197	SCM

(b) Furnace brazing of unplated GlidCop is also acceptable from the joint strength standpoint, if the braze cycle is no more than 2 minutes long.

(c) A copper plating of .02 mm in thickness is adequate for braze time periods of ~ 8 minutes; but it is not adequate if the braze time period is 15 minutes long.

(d) A copper plating of .03 mm is adequate for brazing time periods of up to 15 minutes long. However, the joint strength is slightly reduced when the braze time period is extended beyond 10 minutes.

(e) Joint strengths obtained with Ag-Cu-5% Pd alloy are essentially similar to those obtained with Ag-Cu eutectic alloy.

(f) Direct nickel plated samples showed satisfactory joint strengths, although the metallographic examinations showed that the mode of failure was entirely via the separation of the plated layer from

GlidCop. Inter-diffusion of nickel and copper (or GlidCop) can produce Kirkendal type porosity near plated inter-face. However, at the temperatures commonly used for silver brazing, the size and quantity of pores remain small, provided the braze time period is no more than 15 minutes long.

(g) Samples prepared with a copper strike prior to nickel plating showed higher joint strengths than direct nickel plated samples. Improved adherence of the nickel plating was evident from the observation that failure occurred partially through the GlidCop base material, as shown in Figure 7.

Summary:

1) Successful silver brazing of GlidCop DSC requires electroplating of parts with either nickel or copper, prior to brazing.

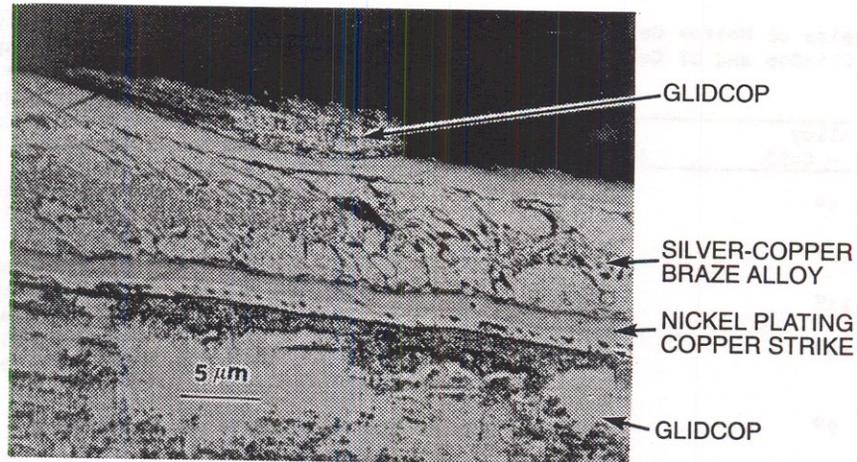


Figure 7: Photomicrograph of a brazed joint (Ag-Cu eutectic) after shear test showing that failure took place partially through the GlidCop base material. Here a copper strike was applied prior to nickel plating to improve adherence.

- 2) Cleaning of parts prior to plating and the use of proper plating techniques are critical.
- 3) The adherence of the plated layer to GlidCop in the case of nickel plating (which is enhanced by a copper strike), and the formation of a smooth and pore free electrodeposit in the case of copper plating, are essential for obtaining sound braze joints.
- 4) It is important that the braze time period be kept as short as possible, so as to prevent excessive inter-diffusion between the nickel plating and GlidCop, and to permit the use of a thinner copper plated layer.
- 5) Firing of the plated parts (or samples) is a means of ensuring the quality of the cleaning and plating practices.

Brazing with Gold-Copper Braze Alloys

Two gold-copper braze alloys were included in this study; namely the Au50-Cu50 alloy and the Au35-Cu65 alloy. Unlike silver, the rate of diffusion of gold along the grain boundaries of copper is similar to its rate of diffusion through the copper grains. In the gold brazing of unplated GlidCop or Copper, one does not experience any preferential diffusion or migration of gold along the grain boundaries. Because of this, a diffusion barrier is not needed when brazing GlidCop with gold-copper braze alloys. Figure 8 is a photomicrograph of a section through a GlidCop-GlidCop braze joint made with Au50-Cu50 alloy. There is no preferential migration of gold along the grain boundaries and there is no void formation of any kind.



Figure 8: Photomicrograph of a braze joint made with Au50-Cu50 alloy showing a narrow and uniform diffusion zone and absence of porosity.

Table 4: Contact Angles of Molten Gold-Copper Braze Alloys on GlidCop and OF Copper Substrates

Substrate Material	Alloy	
	Au35 - Cu65	Au50 - Cu50
OF Copper	9°	10°
AL-15	12°	17°
AL-25	13°	17°
AL-60	13°	18°
AL-15 (Copper Plated)	9°	10°
AL-60 (Copper Plated)	9°	10°

(Note: Hold time-5 minutes, Atmosphere - Hydrogen)

There exists, however, a small difference between GlidCop and copper with respect to the wetting behavior of the gold-copper braze alloys. The gold-copper braze alloys are in general less fluid at their braze temperature compared to the silver-copper braze alloys, and also these do not wet the surface of copper as well as silver-copper braze alloys do. Hence, in the brazing of copper parts with gold-copper braze alloys, one needs to select the braze joint gap carefully in order to achieve a satisfactory joint fill. Pre-placement of braze alloys in the form of foils is usually preferred over the use of wire forms where capillary forces are expected to fill the joint gap. The gold-copper braze alloys do not wet the surface of GlidCop as well as OF copper.

The contact angles of the two gold-copper alloys with GlidCop and with OF copper were determined by performing sessile drop experiments on polished substrates. These experiments were performed in a hydrogen atmosphere, with samples held at 20°C higher than the liquidus of the alloy. The data is shown in Table 4. The contact angles of the two gold-copper braze alloys with GlidCop were larger than with OF copper,

but the angles with copper plated GlidCop were found to be the same as for OF copper. Pre-placement of the braze alloy foil in the joint is most essential when brazing GlidCop with gold-copper braze alloys. However, if the joint configuration does not permit this, then GlidCop samples should be plated with copper to enhance wetting. The copper cyanide bath recommended earlier for silver braze application, is also found satisfactory for gold brazing.

Nickel plating is not recommended when gold brazing GlidCop because a large amount of Kirkendal type pores can develop in GlidCop (or copper) due to excessive inter-diffusion of nickel and the copper matrix at the high temperatures used for gold brazing.

Braze Joint Strengths

The test procedures were similar to those used for the silver braze experiments. All braze temperatures were kept at 10°C above the liquidus of the braze alloy. The shear strengths of joints made with the two braze alloys for the AL-15 and AL-25 grades of GlidCop and various braze parameters were determined. The data for AL-15 is reported in Table 5. The data for AL-25 was similar.

Metallographic examination of the tested specimens showed that in all cases the mode of failure was ductile, and the failure occurred partially through the GlidCop substrate. This indicates that the GlidCop-braze alloy interface and the diffusion zones were at least as strong as the base material. It must be kept in mind here that the yield strength of GlidCop AL-15 is lowered to some extent due to the annealing effects of the high temperature exposure during gold brazing.

Summary:

- 1) GlidCop DSC can be brazed successfully with Au35-Cu65 and Au50-Cu50 braze alloys, but in order to facilitate complete wetting of the substrate one must pre-place the braze foil in the joint. Alternately, the samples may be pre-plated with copper prior to brazing.
- 2) The joint strengths are much higher than those obtained with silver base braze alloys.

Table 5: Shear Strengths of GlidCop AL-15 Brazements Made With Gold-Copper Braze Alloys (L/T=1)

Plating Type	Thickness mm	Braze Alloy	Braze Time	Shear Strength MPa	Data Source
None	--	Au50-Cu50	5	240	SCM
None	--	Au50-Cu50	2	198	Varian
Copper	.020	Au50-Cu50	5	234	SCM
None	--	Au35-Cu65	5	192	SCM
None	--	Au35-Cu65	2	172	Varian
None	--	Au35-Cu65	6	209	Varian
Copper	.020	Au35-Cu65	5	207	SCM

Diffusion Bonding

The primary purpose of the investigation was to determine if GlidCop brand DSC can be joined by the solid-state diffusion bonding process. All three grades of GlidCop were included in the study along with a GlidCop (AL-15) - stainless steel (316) combination. The process parameters (i.e. time, temperature and pressure) used here were the same as is normally used for HIP consolidation of GlidCop powder billets.

Experimental Procedure

All joint configurations were of the lap joint type. Round rods of the sample materials were machined so as to fit together as lap joint specimens. The assemblies were 20 mm in diameter x 250 mm in length. All specimen interfaces were machined to a finish of 125 μ " r.m.s. The assembled specimens were fitted into mild steel tubes which were then evacuated and sealed. The sealed samples were placed in a HIP vessel and HIP'ed for four hours at a temperature of 930°C and a pressure of 107 MPa.

Several rectangular cross-section tensile specimens were machined from each diffusion bonded assembly. Two transverse notches were carefully machined into each specimen to isolate the test area from the rest of the specimen. The distance between the two notches defined the length of the lap joint (L). Two values of L/T were selected (1.4 and 2.0) for all sets of materials. The shear strength value for L/T=1 was extrapolated from a log-log plot of shear strength vs L/T. Table 6 shows the data for all samples.

The experimental data shown here is an average of two tests. Also shown in this table are extrapolated values of shear strength for L/T=1.

Table 6: Joint Strengths of Diffusion Bonded Samples

Material Combination	L/T Ratio	Shear Strength MPa	Elongation (%)
AL-15/AL-15	2.15	149	11
	1.40	211	14
	1.00*	292	--
AL-25/AL-25	2.00	158	6
	1.40	206	8
	1.00*	302	--
AL-60/AL-60	2.00	55	0.5
	1.40	79	0.5
	1.00*	113	--
AL-15/S.S.-316	2.00	123	4
	1.40	151	6
	1.00*	192	--
AL-15 Solid Specimens+	1.40	184	12
	1.00*	235	--
	0.80	270	14
AL-25 Solid Specimens+	1.40	210	10
	1.00*	245	--
	0.80	282	12

(+: Machined to the same configuration as diffusion bonded specimens)
(*: By Extrapolation)

Several GlidCop AL-15 and AL-25 bars were machined to match the geometries of diffusion bonded specimens, annealed, and then tested in shear to determine shear strength of parent material, for comparison. These data are also shown in Table 6.

Results and Discussion

An examination of the data in Table 6 shows that the bond strength was very good in the case of GlidCop AL-15 and GlidCop AL-25. Microscopic examination of the fracture surface showed the mode of failure to be ductile, and also that the failures took place only partially through the bonded interface. The relatively high tensile elongations of the specimens indicate that the joints possessed fairly high toughness. The GlidCop AL-15 and stainless steel combination also showed adequate bond strengths and the mode of failure here was also ductile.

The GlidCop AL-60 specimens did not develop satisfactory bonding in this initial effort. It is presumed that a smoother surface finish would lead to greater interface contact and help achieve satisfactory bonding. The use of a higher HIP'ing pressure could possibly help improve bonding. Work with GlidCop AL-60 grade continues.

Summary:

1) The AL-15 and AL-25 grades of GlidCop DSC can successfully be joined by diffusion bonding. These grades develop satisfactory bond strengths, under the pressure, temperature and time parameters that are normally used for HIP'ing.

- 2) GlidCop AL-15 can also be diffusion bonded satisfactorily to stainless steel (316).
- 3) GlidCop AL-60 will likely require a smoother surface finish, coupled with a higher HIP pressure, to develop sufficient bond strength.

Acknowledgements

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Appendix I: Cleaning of GlidCop for Electroplating

Listed here are four separate cleaning procedures that are used by individual electroplaters to clean GlidCop DSC prior to electroplating. These procedures are their standard practices for cleaning copper and are found to be effective for cleaning GlidCop as well.

However, since GlidCop has a much finer grain structure compared to copper, the likelihood of the residues of the cleaning solutions remaining at the grain boundaries is much greater. Entrapped residues of the cleaning agents (or any precipitates formed by the combination of two or more cleaning agents) cause the electroplating to develop blisters afterwards, during brazing. Therefore, it is important that all rinsing steps are carried out thoroughly.

Procedure-A:

1. Solvent degrease in trichloroethane.
2. Rinse well.

3. Alkaline-soak clean in a commercially available alkaline soak (NaOH) solution, such as UDYPREP[®] 126. UDYPREP[®] 126 is manufactured by OMI/UDYLITE, 21411 Hoover Rd., Warren, Mich. 48089, Tel No. (313) 497-9100.
4. Rinse well.
5. Dip in Hydrochloric Acid (reagent grade, 37% HCl), for 30 to 40 seconds.

6. Rinse well.

Procedure-B:

Steps 1 thru 6 are same as in Procedure A.

7. Bright dip in a mixture of Conc. HNO₃ (1 part) and Conc. H₂SO₄ (2 parts).
8. Rinse well in tap water first and then in deionized water.

Procedure-C:

1. Solvent degrease in trichloroethane.
2. Rinse well.
3. Alkaline-soak clean in BroCo HDM, a commercially available caustic solution available from BroCo Products, Inc., 18624 Superior Ave., Cleveland, Ohio 44110, Telephone No. (216) 531-0880.
4. Rinse well in free flowing water. If grooves or holes are present in the part, use an ultrasonic bath for additional rinsing.
5. Bright dip in a mixture of conc. HNO₃ (1 part) and conc. H₂SO₄ (2 parts).
6. Rinse well.

Procedure-D:

Steps 1 thru 4 are same as in Procedure A

5. Dip parts for 20 seconds in "Copper Etching Solution", which is made up as follows: Mix carefully 75% by volume nitric acid (70% HNO₃) and 25% by volume phosphoric acid (85% H₃PO₄). Bath should be at room temperature.
6. Rinse in cold running water for one minute.
7. Immerse in "Copper Bright Dip" solution and hold there for two minutes. Copper Bright Dip solution is made up as follows: Mix carefully 55% by volume phosphoric acid (85% H₃PO₄), 20% by volume nitric acid (70% HNO₃) and 25% by volume glacial acetic acid (99.7% CH₃COOH).
8. Rinse in cold running water for 15 seconds.
9. Rinse in hot running water for 15 seconds.
10. Rinse in cold running water for 2 minutes.
11. Clean in Isopropyl Alcohol, using an ultrasonic bath, for 2 minutes.

is: $\text{CuCN} - 15 \text{ g/l}$, $\text{NaCN} - 28 \text{ g/l}$ and $\text{Na}_2\text{CO}_3 - 15 \text{ g/l}$, with Cu as metal being 10.5 g/l and free NaCN being 11 g/l . The cathode current density should be about 200 A/m^2 . The bath temperature should be 50°C to 63°C , and the plating thickness should be 2 to 3 microns.

B. Copper Cyanide Bath: Reliable platings can be produced from a commercially available plating process known as E-Brite 23-11, marketed by Electrochemical Products, Inc. (EPI), 17000 Lincoln Ave., New Berlin, Wisconsin 531512, telephone: (414) 786-9330. The bath composition is, Cu as metal - 41 g/l , Free KCN - 13.5 g/l and KOH - 15 g/l . Operating temperature is 65°C and current density is 400 A/m^2 . [Other copper cyanide baths, such as one comprising of 2 moles of NaCN for each mole of CuCN, are also found to produce satisfactory deposits.]

C. Copper Sulfate Bath: The bath composition is, Copper Sulfate - 188 g/l (Cu, as metal - 48 g/l) and Sulfuric Acid - 75 g/l . The bath temperature should be between 32°C and 43°C and the cathode current density should be between $300-500 \text{ A/m}^2$.

D. Watts Nickel Bath: Nickel plating should be done in a dull Watts Bath (Acidic). The bath should be free from impurities (especially chromium) and brighteners. The bath composition is as follows:

Nickel Sulfate -- 285 g/l
Nickel Chloride -- 50 g/l
Boric Acid -- 38 g/l
Total Nickel
as metal -- 85 g/l
pH -- 3.8 to 4.0 (critical)
Temperature -- 60°C
Current Density -- 300 A/m^2
Antipitting Agent -- 0.5%, by volume

Antipitting agent recommended is made by Udylite Corp., Detroit (Mich.), called "Anti-Pitter 22 Special", and it does both antipitting and wetting. Telephone No. of Udylite Corp. is (313) 497-9100.

The pH of the electrolyte should be closely monitored. If the pH becomes greater than 4.0, the throwing power is adversely affected and if it goes to below 3.8, the deposit becomes stressed. The current source for plating should provide a fairly "flat" D.C. current, (i.e. with little or no ripples.)

Only high purity nickel anodes should be used and anodes should be "bagged" to retain particulate contaminants.

