ANALYSIS OF RING AND PLUG SHEAR STRENGTHS FOR COMPARISON OF LEAD FREE SOLDERS

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ABSTRACT

The drive to replace the use of toxic lead metal and its alloys has spurred the development of many new Lead-free solder alloys. In addition to the toxicity of lead, there are other problems concerning the mechanical properties of Sn-Pb and Pb-based solders. Moreover, current leaded solders lack shear strength, resistance to creep and to thermal-mechanical fatigue. A solder which exhibits enhancements of these properties and retains solderability is crucial in avionics, automotive electronics, and industrial applications where the solder joints are subjected to many thermal cycles, severe vibrations, and sustained temperatures of up to 125˚C. Modified ring and plug joints were made with 18 selected lead-free solders and 3 well characterized lead-containing solders. The results of the mechanical tests under varying temperature and strain rate conditions provide a basis for selection of the optimum lead free solder for elevated temperature applications.

INTRODUCTION

The relatively recent drive to replace the use of toxic lead metal and its alloys in industrial applications has spurred the development of new Pb-free solder alloys[1]. In addition to the toxicity of lead, the mechanical properties of Sn-Pb and Pb-based solders are often lacking at elevated temperatures. Current leaded solders exhibit low shear strength and resistance to creep and to thermal-mechanical fatigue[2]. A solder which exhibits enhancements of these properties and retains solderability is crucial in avionics, automotive, and industrial applications where the solder joints are subjected to many thermal cycles, severe vibrations, and sustained temperatures of up to 125˚C. The consequences of solder joint failure in these critical applications or in any application where "lifetime" performance is expected can be disastrous. In addition to improved mechanical properties a viable lead-free solder alloy should have similar processing characteristics to Sn-Pb solders. Solder alloys that require major modification or replacement of existing soldering equipment to accommodate higher melting temperatures would not be favored[1].

The shear strength of a solder joint is very important parameter in the selection of a new solder composition, because the joint interface microstructure along with the bulk solder microstructure is tested. One method that is commonly used to measure shear strength of solders is the ring and plug test[3]. Although this test is accepted as a shear test that provides reliable and consistent results, varying shear strengths are sometimes obtained even for similar conditions[4].

In addition, different shear test configurations can produce disparate results for the similar processing and testing conditions[3-4]. There is some indication that some of the variability observed is due to voids or porosity formed in the shear test joint[4]. Having a test that produces consistent results can help determine what alloy or alloys would be suitable for further production testing.

EXPERIMENTAL PROCEDURE

The twenty-one alloys investigated in this study are listed in weight percent in Table 1. It should be noted that all compositions are listed in weight percent. The alloys were manufactured by Johnson Manufacturing with their standard industrial alloy practice. The lead-free alloys were selected to cover a fairly broad range of lead-free compositions and taking the final application into account. As this work was originally conceived to test lead-free radiator solders, the first twelve alloys are sensible for structural applications. The next nine alloys exhibit phase equilibria more conducive to electronic packaging.

Table 1 Nominal Composition (weight %) of Alloys Studied

ID	Sn	Cu	Ag	Bi	Sb	Zn	Ni	Pb	Te	Co
1	98.5	1.5	-	-	-	-	-	-	-	-
2	97.0	3.0	-	-	-	-	-	-	-	-
3	95.5	4.0	0.5	-	-	-	-	-	-	-
4	95.0	-	-	-	5.0	-	-	-	-	-
5	91.0	-	-	-	-	9.0	-	-	-	-
6	97.0	2.0	0.2	-	0.8	-	-	-	-	-
7	98.4	0.1	0.1	-	1.3	-	0.1	-	-	-
8	91.8	-	0.2	-	-	8.0	-	-	-	-
9	97.25	0.375	0.375	-	2.0	-	-	-	-	-
10	93.5	5.0	0.5	0.5	-	-	-	-	0.5	-
11	30.0	-	-	-	-	-	-	70.0	-	-
12	3.0	-	1.5	-	-	-	-	95.5	-	-
13	96.35	-	3.65	-	-	-	-	-	-	-
14	93.6	1.7	4.7	-	-	-	-	-	-	-
15	95.25	1.0	3.6	-	-	-	-	-	-	0.15
16	95.5	0.7	3.8	-	-	-	-	-	-	-
17	96.2	0.8	2.5	-	0.5	-	-	-	-	-
18	91.84	-	3.33	4.83	-	-	-	-	-	-
19	99.3	0.7	-	-	-	-	-	-	-	-
20	42	-	-	58	-	-	-	-	-	-
21	63	-	-	-	-	-	-	37	-	-

The ring and plugs were machined to the dimensions illustrated in Figure 1. The joint gap of 175 microns was chosen after several tests by Johnson Manufacturing showed that this gap was less likely to produce microporosity of the lead-free alloys. The machined ring and plugs were cleaned using an acid etch (ITRI formula) and rinsed thoroughly with DI water, then with alcohol. A preform of solder was made for each ring and plug sample. Flux (Johnson's #1) was applied by dipping all parts in a beaker containing flux. The assembled ring and plug were moved to a hot plate where additional flux was added with a brush as needed. The samples were heated to a maximum temperature of 350˚C at about 1˚C/sec to ensure that a solid joint was made. After soldering, the samples were placed on a heat sink to cool to room temperature at about 1.5˚C/sec. It should be noted that spacing wires were not used in this work, because of the problems they appeared to induce in the sample joint integrity. Moreover, the relatively high soldering temperature appeared to be required to obtain a completely solid joint. Although, the lack of spacing wires did produce some joints with varying thickness, the results obtained were relatively consistent. The samples were tested using the test fixture pictured in Figure 2. The test fixture ensured a consistent set up was achieved during each test. In addition, the test fixture reduced the risk of having off axis loading occur. Three samples of each of the compositions listed in Table 1 were tested at room temperature at a displacement rate of 0.1 mm/min. After the room temperature shear tests, six alloys were chosen from the original twelve structural solders for additional testing. Three test samples of each of the six structural alloys and the nine electronic packaging alloys were tested at 125˚C with a displacement rate of 0.1 mm/min. In addition, three test samples of each of the six structural alloys were tested at -76˚C with a displacement rate of 0.1 mm/min and at room temperature at 10 mm/min. Samples of the electronic packaging alloys were sectioned, mounted and polished for metallographic examination.

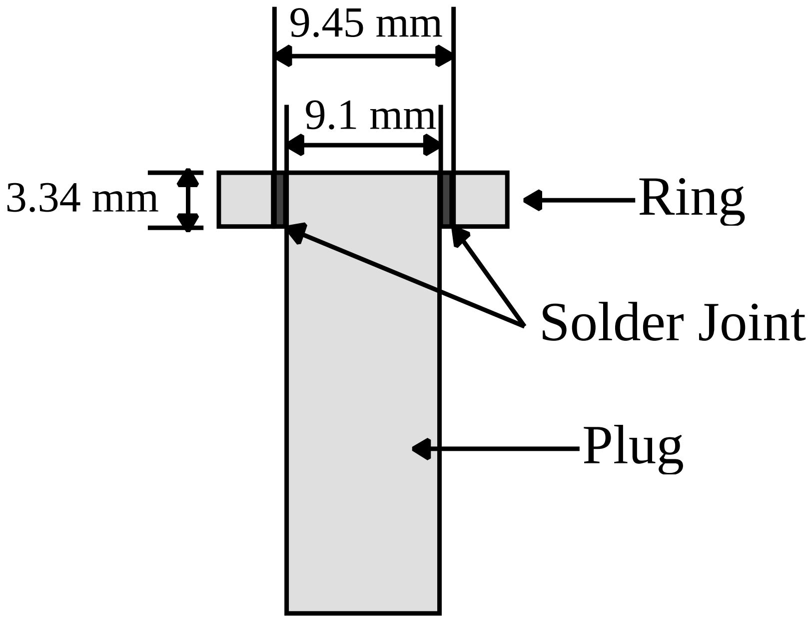

Figure 1. Diagram of the ring and plug sample configuration used in this study.

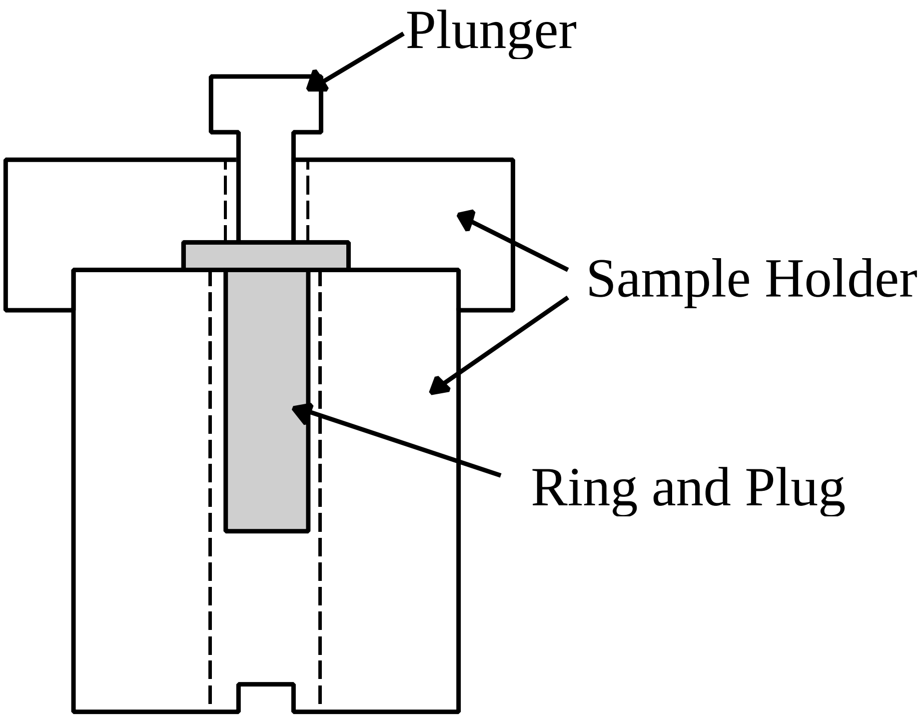

Figure 2. Diagram of the ring and plug holder used in this work.

RESULTS

The average shear strength and standard deviation of three separate tests of each of the compositions listed in Table 1 tested at room temperature are listed in Table 2. The shear strength values vary from 22.7 MPa for alloy 12 (Pb-3Sn-1.5Ag) to 64.4 MPa for alloy 18 (Sn-3.33Ag-4.83Bi). In addition, except for alloys 8, 17, 18 and 20, the results obtained exhibited very limited variability within each test group.

The average shear strength and standard deviation of the three test samples of each of the six structural alloys and the nine electronic packaging alloys tested at 125˚C with a strain rate of 0.1 mm/min are listed in Table 3. The shear strength values range from 6.8 MPa for alloy 20 to 26.2 MPa for alloy 18. The results of these tests exhibit even less variability than the tests performed at room temperature. Alloys 13 and 18 exhibited the greatest high temperature (125˚C) standard deviation of 1.5 MPa and 2.7 MPa respectively.

The average shear strength and standard deviation of the three test samples of each of the six structural alloys tested at room temperature with a strain rate of 10 mm/min are listed in Table 4. The shear strength values range from 44.6 MPa for alloy 2 to 55.0 MPa for alloy 10. The average shear strength and standard deviation of the three test samples of each of the six structural alloys tested at -76˚C with a strain rate of 0.1 mm/min are listed in Table 5.

Table 2. Average shear strength and standard deviation of tests conducted at 0.1 mm/min at room temperature.

Sample ID	Shear Strength (MPa)	Standard Deviation
1	27.4	±0.4
2	28.9	±0.6
3	32.0	±0.8
4	31.6	±1.1
5	28.8	±0.9
6	35.5	±0.7
7	33.5	±0.9
8	28.3	±5.2
9	36.5	±0.1
10	37.0	±0.5
11	26.9	±1.3
12	22.7	±0.7
13	37.2	±1.2
14	40.5	±0.7
15	42.9	±0.8
16	35.1	±0.8
17	35.1	±3.5
18	64.4	±4.8
19	27.0	±1.0
20	46.2	±2.9
21	32.7	±0.9

Table 3. Average shear strength and standard deviation of tests conducted at 0.1 mm/min at 125˚C

Sample ID	Shear Strength (MPa)	Standard Deviation
2	13.8	±0.9
4	14.2	±1.1
5	13.8	±1.4
7	12.8	±0.5
8	12.7	±0.9
10	12.0	±1.2
13	18.4	±1.5
14	17.2	±0.6
15	20.8	±1.5
16	18.2	±0.5
17	15.7	±1.1
18	26.2	±2.7
19	13.3	±0.4
20	6.8	±0.6
21	12.2	±1.2

Table 4. Room temperature 10 mm/min shear strength tests results

Sample ID	Shear Strength (MPa)	Standard Deviation
2	44.6	±2.2
4	52.1	±1.1
5	48.2	±3.8
7	51.8	±0.6
8	52.2	±0.7
10	55.0	±2.6

Table 5. Average shear strength and standard deviation of tests conducted at 0.1 mm/min at –76˚C.

Sample ID	Ranking (-76˚C)	Shear Strength (MPa)	Standard Deviation
2	4	64.6	±3.3
4	5	64.2	±8.0
5	3	68.2	±2.0
7	2	74.0	±1.9
8	1	77.0	±1.5
10	6	57.7	±12.8

Representative microstructures for samples 13-21 are shown in Figures 3-11 respectively. Except for the microstructures shown in Figures 3 and 4, all are representative of untested joints. Moreover, the upper joint surface is that of the plug and the lower is the ring surface. Notice that voids caused by mechanical testing are observed closer to the smaller inner joint surface of Figures 3 and 4. The microstructures typically consist of Cu_6Sn_5 intermetallics and in some cases Cu_3Sn (darker than Cu_6Sn_5) growing from the copper interface. The extent of Cu_6Sn_5 growth varies: relatively long for Sn-0.7 (Figure 9) to relative short Cu_6Sn_5 for Sn-58Bi (Figure 10). Moreover, the microstructure present in the inner joint region varies from a simple eutectic without intermetallics (Figures 10 and 11) to microstructures that contain a relatively large amount of Cu_6Sn_5 and Ag_3Sn.

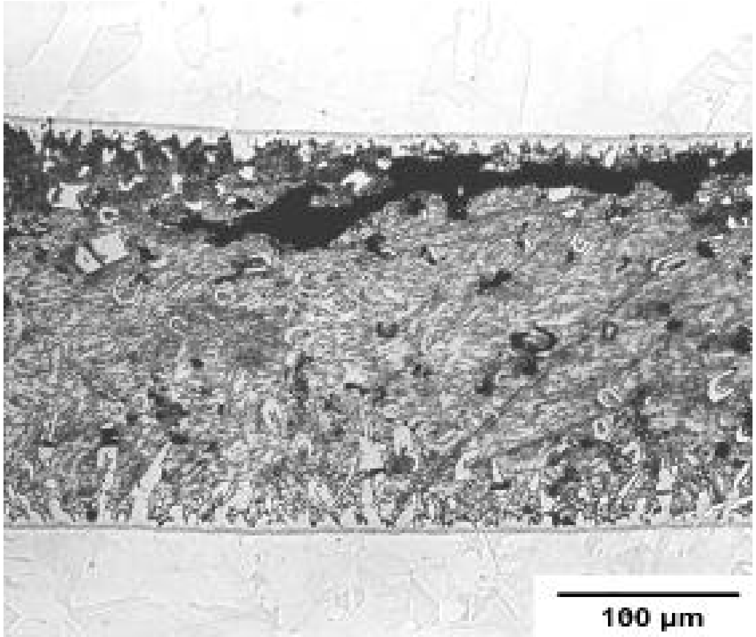

Figure 3. Optical micrograph of a representative Sn-3.65Ag ring and plug joint after failure.

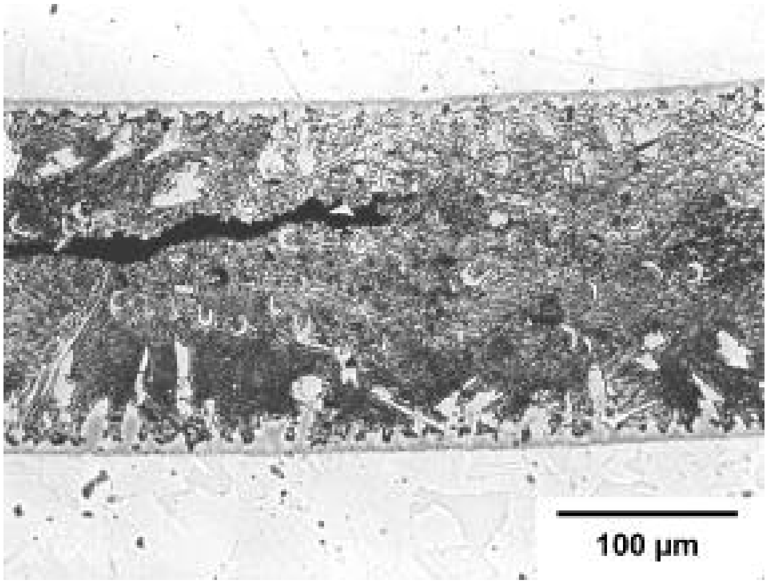

Figure 4. Optical micrograph of a representative Sn-4.7Ag-1.7Cu ring and plug joint after failure.

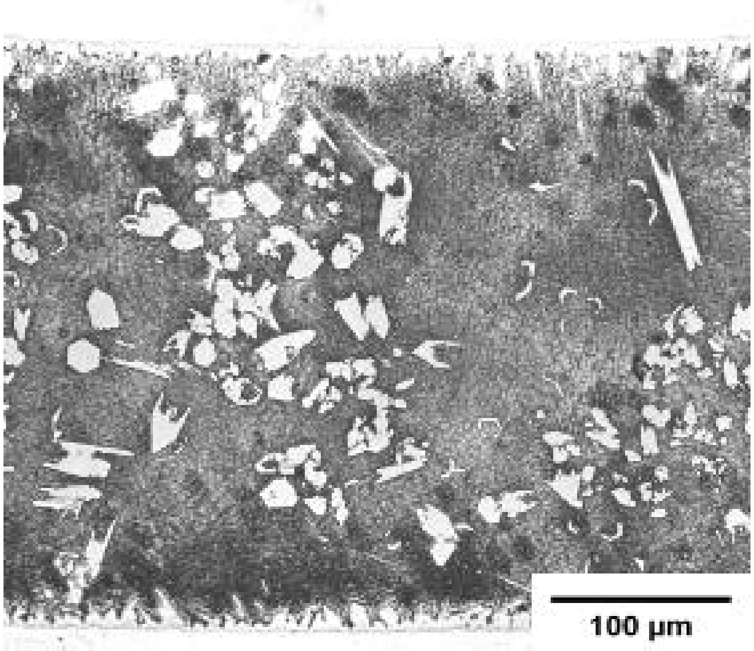

Figure 5. Optical micrograph of a representative Sn-3.6Ag-1Cu-0.15Co ring and plug joint.

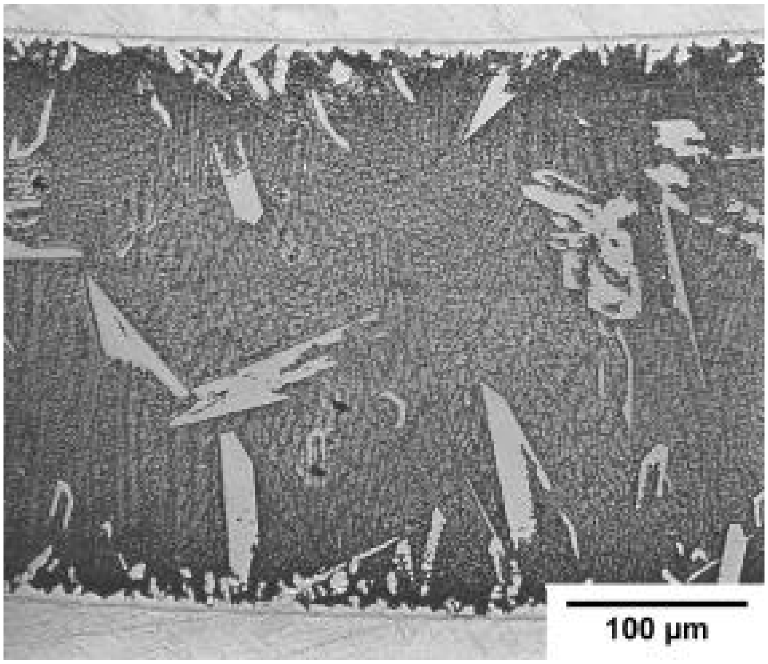

Figure 6. Optical micrograph of a representative Sn-3.8Ag-0.7Cu ring and plug joint.

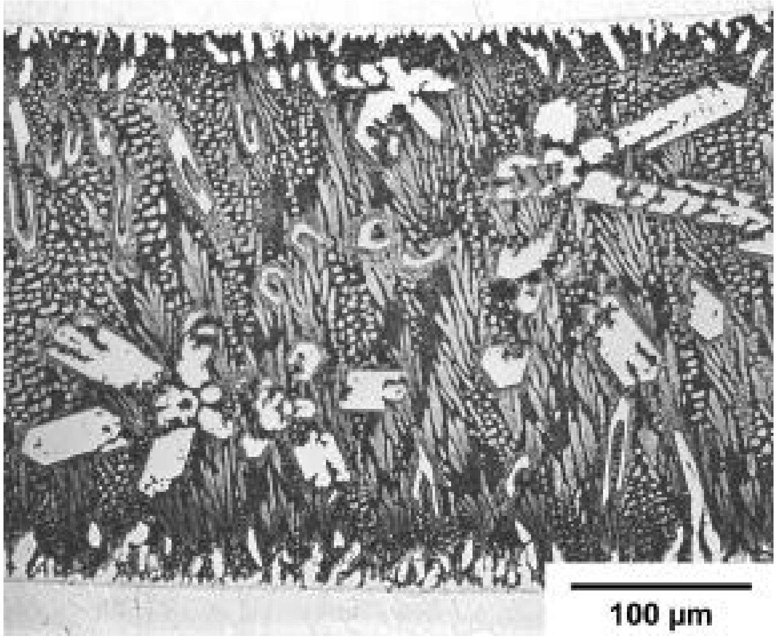

Figure 7. Optical micrograph of a representative Sn-2.5Ag-0.8Cu-0.5Sb ring and plug joint.

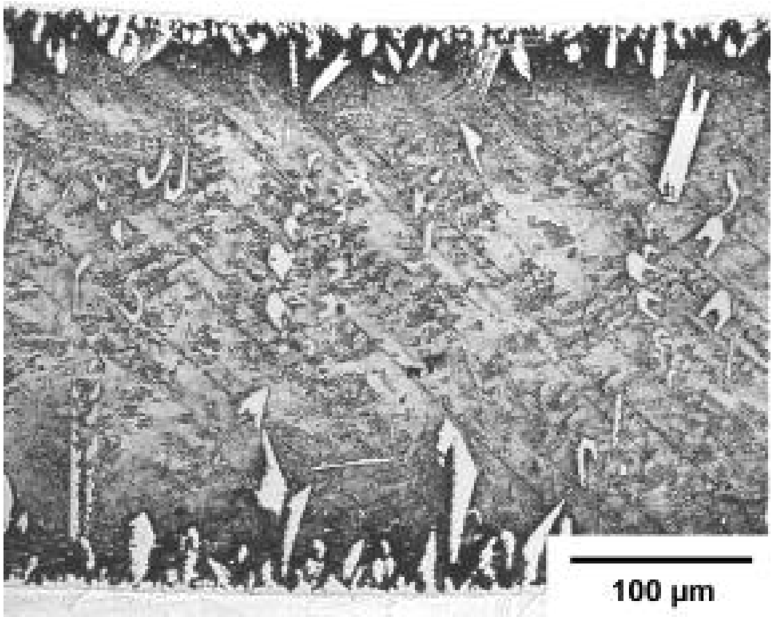

Figure 8. Optical micrograph of a representative Sn-3.33Ag-4.83Bi ring and plug joint.

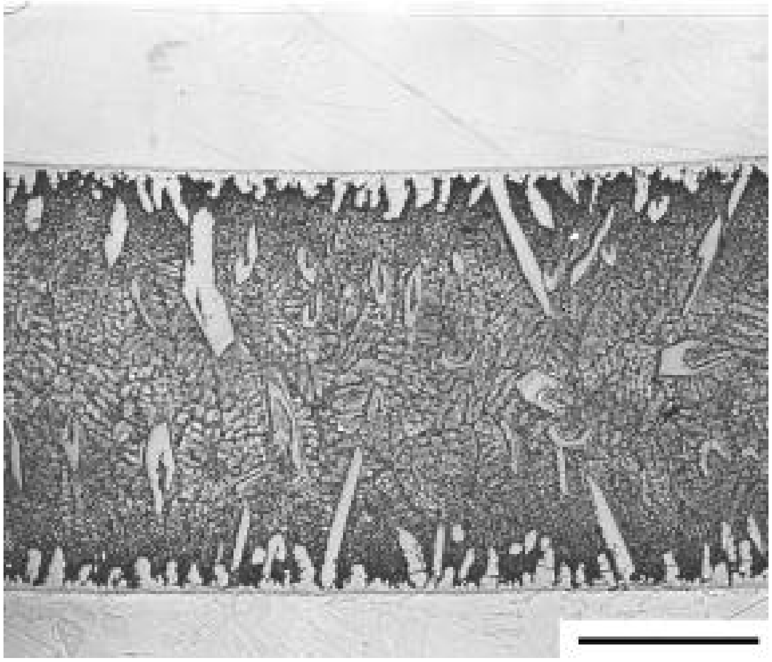

Figure 9. Optical micrograph of a representative Sn-0.7Cu ring and plug joint.

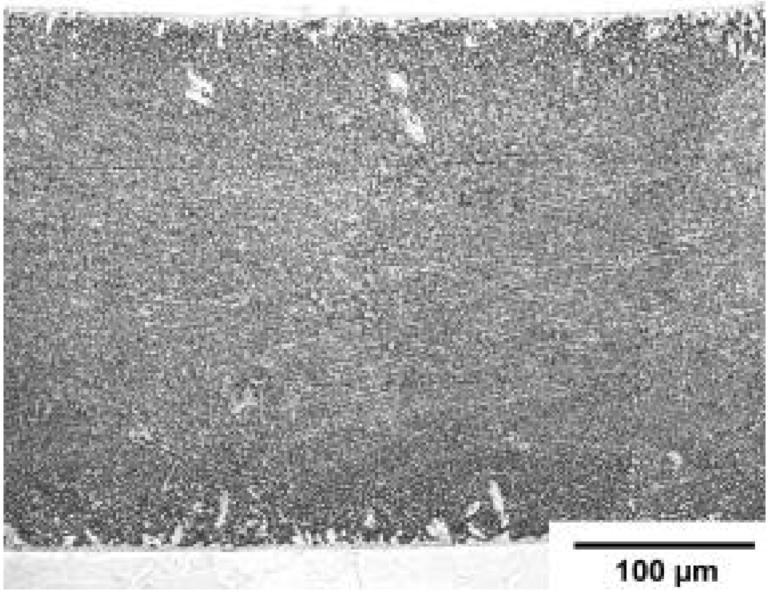

Figure 10. Optical micrograph of a representative Sn-58Bi ring and plug joint.

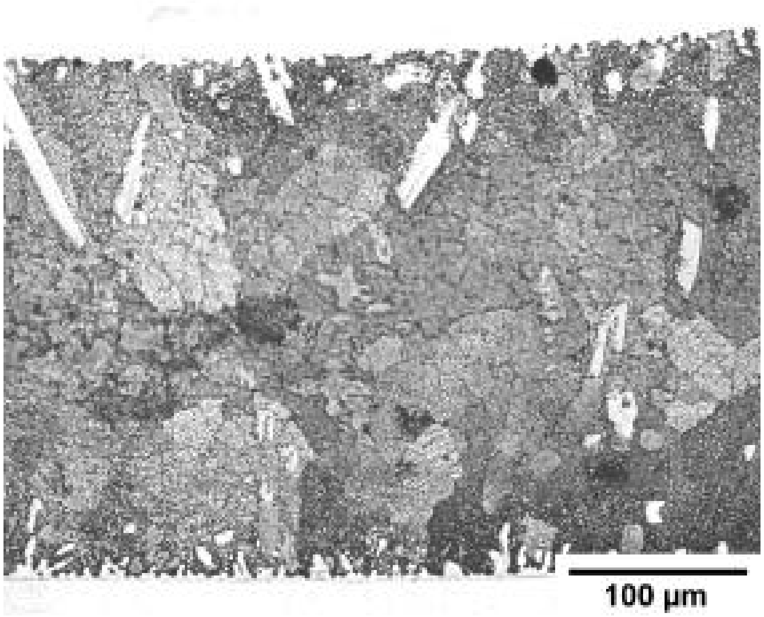

Figure 11. Optical micrograph of a representative Sn-37Pb ring and plug joint.

DISCUSSION

The results obtained from the use of the modified ring and plug configuration provided very consistent and dependable results. The 125˚C tests appear to produce very limited variation in measured shear strength compared to the room temperature tests. The consistent results along with observations that ring and plug samples made with wires had a higher tendency to produce voids (examples shown in Figure 12) bring into question the use of wires as spacing guides. The wires that were attempted to be used, before samples were made without them, in this work were the International Tin Research Institutes (ITRI) recommended nichrome wires. Other researchers[5], have reportedly used copper spacing wires instead of nichrome, presumably to reduce the voiding tendency encountered when nichrome was used. Regardless, the presence of wires in the joint to be tested is something that is not desired, but tolerated to obtain a consistently thick joint[6]. The results obtained from this study indicate that they are unnecessary. Moreover, in the case of structural applications, the observed non-concentricity is characteristic of the joints formed during normal production and therefor the tests are more a measure of how production joints will perform in service.

Figure 12. Examples of plugs that were not wet by the solder when spacing wires of nichrome were used.

The very reasonable variation from run to run and the not very consistent joint concentricity from sample to sample, indicate that the joint integrity obtained was very consistent. Moreover, the metallographic inspection of the joints indicates that the samples examined in this study did not have significant amounts of porosity. Further supporting evidence of consistent joint integrity is the apparently higher mechanical strength obtained in this study for Sn-3.5Ag (37.3 MPa this study) and what is reported (27 MPa) by other solder manufactures[5] using the ring and plug sample configuration at the same strain rate and temperature.

With one exception, the room temperature tests performed at a displacement rate of 0.1 mm/min indicate that all of the lead-free alloys tested exhibit higher shear strength values than two of the three Pb containing alloys (alloy 11 and 12) tested. Alloy 19 (Sn-0.7Cu) exhibited a shear strength of 27.0 MPa that is equal to the shear strength of alloy 11 when the standard deviation is taken into account. It should be noted that alloy 21 exhibited a very respectable (32.7 MPa) shear strength value, when compared to a reported value (23 MPa)[5]. Again, the higher shear strength values obtained in this study may be due to the formation of a completely void free joint and a contiguous joint interface. Moreover, the obtained result may be a more realistic measure of the true shear strength.

The alloys that exhibited the highest room temperature strength were Sn-3.33Ag-4.83Bi (64.4 MPa) and Sn-58Bi (46.2MPa). It is apparent that Bi is a very potent shear strengthener of Sn. Moreover, the relatively large difference between Sn-3.33Ag-4.83Bi and Sn-58Bi indicate that Ag is also a very good strengthening addition to Sn. It should also be noted that the Sn-58Bi micrograph shown in Figure 10 does not exhibit a large amount of intermetallic anywhere in the joint. The lack of intermetallics and relatively high shear strength suggests that the intermetallic observed in weaker solder joints are responsible for the lower strength. This implies that, the morphology of the intermetallic at the joint interface doesn't have a strong effect on the measured shear strength.

The results of the 125˚C tests indicate that higher temperatures, as expected, reduce the shear strengths that are observed. Some alloys retained more of the room temperature shear strength than others did. Retention of room temperature strength is an indication how stable the joint is under varying thermal conditions. The difference in behavior is illustrated by dividing the high temperature (125˚C) strength by the room temperature strength to obtain a percentage. A plot of the calculated percentage for each alloy is shown in Figure 13. It should be noted that the shear strength of a relatively strong lead-free solder (Alloy 20 Sn-58Bi) exhibited a very poor shear strength at 125˚C. Sn-37Pb retained more of its room temperature strength than the Sn-58Bi did. The low high temperature shear strength is due to the relatively low melting temperature (138˚C) of this alloy. Use of Sn-58Bi at temperatures around 125˚C and above would not be advisable.

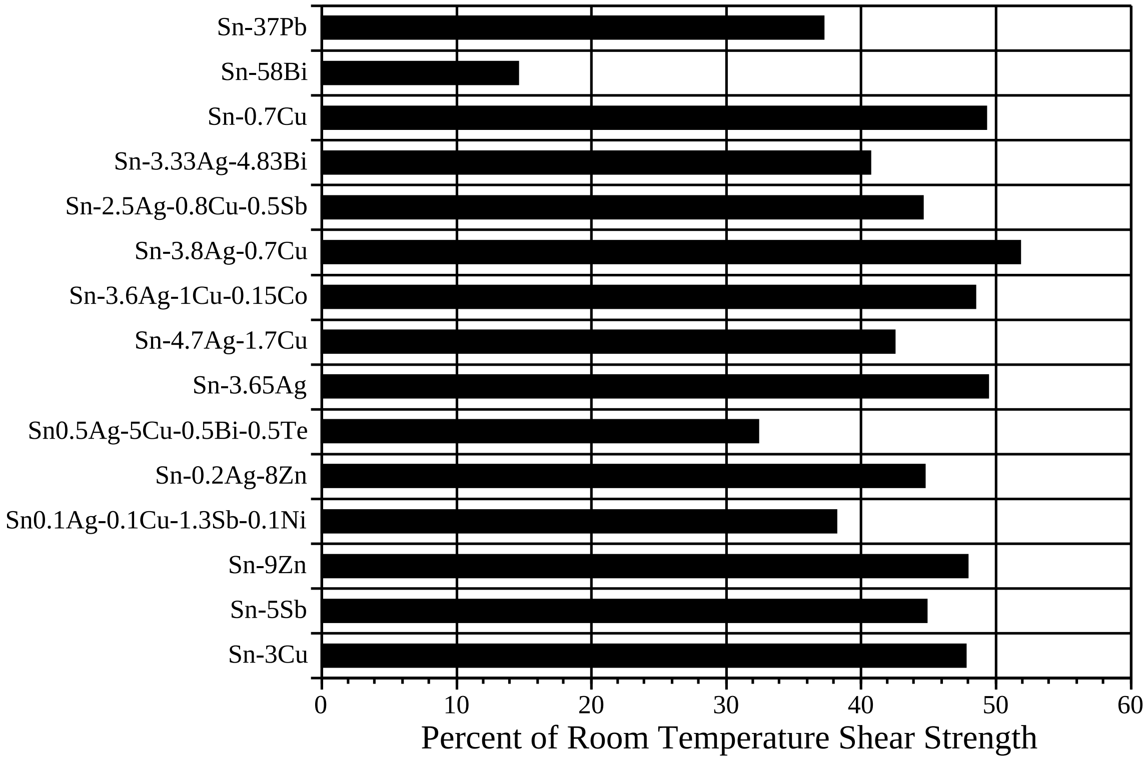

Figure 13. Graph of retained room temperature shear strength at 125˚C for all the solders tested.

Alloys that did retain nearly 50% or more of the room temperature strength at 125˚C were Sn-3.8Ag-0.7Cu, Sn-0.7Cu, Sn-3.65Ag, and Sn-3.6Ag-1Cu-0.15Co. It is interesting to note that except for the Sn-0.7Cu alloy the level of silver is very similar. Moreover, except for Sn-3.65Ag alloy the level of copper is very similar. This would seem to indicate that additions of silver and copper produce a microstructure that is desirable for retaining room temperature shear strength. However, it should be noted that the Sn-4.7Ag-1.7Cu alloy retained only about 44% of its observed room temperature shear strength indicating that larger amounts of Ag and Cu do not continue to contribute to retention of room temperature shear strength at higher levels.

It should also be noted that the Sn-3.33Ag-4.83Bi alloy, while still exhibiting the highest shear strength only retained about 40% of the room temperature strength. The greater loss of room temperature strength is an indication that the Bi is not as potent a strengthener at elevated temperatures. Moreover, previous results indicate that a small addition of Cu may improve shear strength retention. Further work is required though to verify this hypothesis.

CONCLUSIONS

The modified ring and plug specimens produced in this work provided consistent and reliable measurements of the shear strength of the alloys tested. All of the modified ring and plug specimens exhibited good wetting in contrast to specimens made with spacer wires. A lack of concentricity of the joints did not appear to affect the shear strength measurements. Sn-3.33Ag-4.83Bi exhibited the highest room temperature and high temperature (125˚C) shear strength of all the alloys tested. Sn-0.7Cu exhibited the lowest room temperature shear strengths. Bi additions appear to dramatically increase the room temperature shear strength, but elevated (125˚C) temperatures significantly decrease strengthening affect. From the alloys studied, Ag additions appear to be the next best addition followed by Cu. No correlation could be made between the optical micrographs and shear strength measurements.

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