A study of solder alloy ductility for cryogenic applications

A. Lupinacci[1], A. A. Shapiro[2*], J-O. Suh[2], A.M. Minor[1]

[1]Department of Materials Science and Engineering, University of California, Berkeley and National Center for Electron Microscopy, Lawrence Berkeley National Laboratory, Berkeley, CA 94720

[2]Jet Propulsion Laboratory, California Institute of Technology, 4800 Oak Grove Drive, Pasadena, CA 91109 USA

*Corresponding Author: Andrew.A.Shapiro@jpl.nasa.gov

Abstract

For aerospace applications it is important to understand the mechanical performance of components at the extreme temperature conditions seen in service. For solder alloys used in microelectronics, cryogenic temperatures can prove problematic. At low temperatures Sn-based solders undergo a ductile to brittle transition that leads to brittle cracks, which can result in catastrophic failure of electronic components, assemblies and spacecraft payloads. As industrial processes begin to move away from Pb-Sn solder, it is even more critical to characterize the behavior of alternative Sn-based solders. Here we report on initial investigations using a modified Charpy test apparatus to characterize the ductile to brittle transformation temperature of nine different solder systems.

1. Introduction

As aerospace applications such as planetary and manned space exploration continue to push the limits of materials both in thermal requirements and long-term reliability, it is even more necessary to understand the failure modes of these materials. Space applications demand high reliability over a wide range of temperatures. For example, the space environment can cause equipment to get as hot as 100C and as cold as -230C with the change in temperature occurring in 10's of seconds or minutes [1,2]. Table I gives several examples of typical thermal requirements for spacecraft components.

Table I: Examples of typical thermal requirements for spacecraft components [1].

Component	**Typical Temperature Ranges (C)**	
	Operational	**Survival**
Batteries	0 to 15	-10 to 25
Power Box Baseplates	-10 to 50	-20 to 60
Reaction Wheels	-10 to 40	-20 to 50
Gyros/IMUs	0 to 40	-10 to 50
Antennas	-100 to 100	-120 to 120
Solar Panels	-150 to 110	-200 to 130

Solder is used to make mechanical, thermal and electrical connections for electronic components used in a wide variety of space applications such as printed wiring boards and solid state power amplifiers. On-orbit thermal cycling can not only embrittle the solder as it crosses the ductile to brittle transition temperature (DBTT), it can fatigue the joint, bringing into question the long-term reliability of solder joints at cryogenic temperatures. Planetary or Lunar environments can be as low as -230°C. The surface of Saturn's moon Titan is very stable at around -180°C. Missions to Titan and other extreme environments are have been recently proposed by the National Research Council's Decadal Survey [3]. Reliability of any solder joint interconnection directly depends on the resilience of the joint structure and material to the imposed stress due to thermal mismatch of the mating part or material [4].Therefore, it is critical to evaluate current and proposed solder alloys for their reliability in the expected extreme environments for future space missions.

Of particular interest to this study is the failure mode of solder at cryogenic temperatures. At low temperatures, Sn-based solders can undergo a ductile to brittle transition that leads to brittle cracking and subsequently, catastrophic failure of electronic components. As the aerospace industry begins to move away from Pb-containing solder, it is even more necessary to understand the low-temperature behavior of alternative Sn-based solders.

A potential reliability issue for solder alloys used in low temperature applications is the allotropic phase transformation of Sn, which is also commonly known as the 'tin pest'. Pure Sn undergoes allotropic phase transformation from β-Sn (white tin) to α-Sn (grey tin) around 13°C. During the phase transformation, a large amount of volume change (~27%) takes place, causing localized rupture of the solder. Once formed, the α-Sn can exist at temperatures up to around 60°C. The allotropic phase transformation takes place by nucleation and growth process. The nucleation process has a prolonged incubation period ranging from several months to decades. This incubation time can be significantly reduced by inoculating seeding materials at the surface of a bulk β-Sn. The seeding material can be α-Sn or any material with a diamond cubic crystal structure with lattice parameter close to 6.4Å, such as CdTe, InSb, or metastable ice [5,6]. Figure 1 (a) and (b) show bulk β-Sn samples inoculated with InSb particles. The sample in the figure (a) was stored in room temperature and the sample in the figure (b) was stored at -25°C for 1 day and -35°C for 3 days. The α-Sn has propagated throughout the entire surface, causing ruptures. The α-Sn can propagate as fast as 3mm/hr after the inoculation. The growth rate depends on temperature and dimension of the specimen. The thicker samples tend to exhibit faster growth rates, and the growth rate is highest around -20°C to -40°C [7]. This is a temperature range that spacecraft and avionics electronics frequently encountered. The prolonged exposure of a solder alloy to such temperatures can cause failure of solder joints by the allotropic transformation, especially if the electronics are exposed to materials such as CdTe or InSb during the assembly or naturally form metastable ice in the atmosphere during the operation.

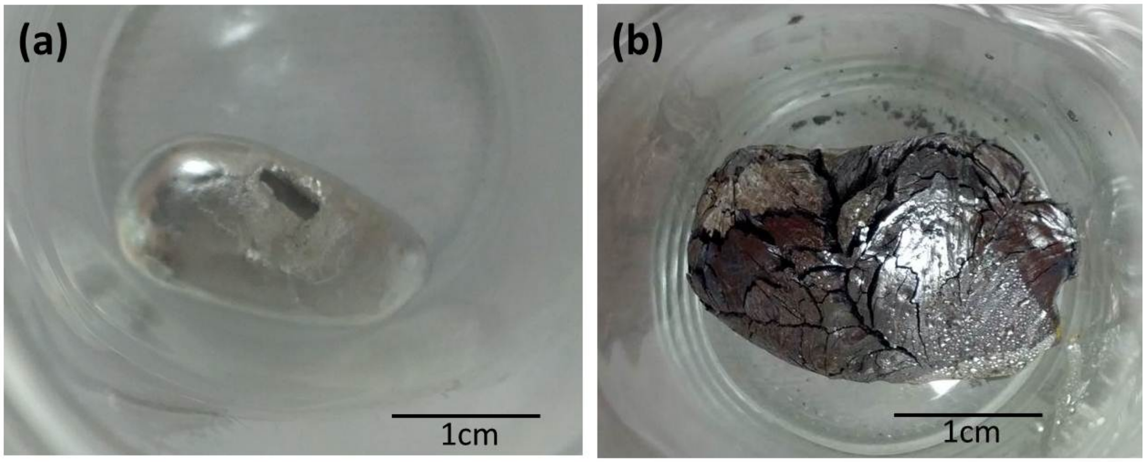

Figure 1. Bulk Sn samples inoculated with InSb particle. (a) Stored in room temperature, (b) stored at at -25°C for 1 day and -35°C for 3 days.

Importantly, the mechanical properties of Sn-based solders have not been adequately characterized at cryogenic temperatures and the mechanisms are not well understood. Therefore, this study aims to map the DBTT for various solder systems. Future work will focus on both the DBTT, the allotropic phase transition and deformation mechanisms at low temperatures for Sn-based solder alloys.

2. Experimental

This study used a modified Charpy test as described by Zachrisson, et al. [8]. This study focused on six different bulk solder systems, summarized in Table II. A minimum of 25 Charpy specimens were tested for each alloy. Figure 2 illustrates the shape and size of the Charpy specimen used for each alloy. The temperature for each alloy was established through a series of heating curves that were measured prior to Charpy testing. The heating curves were established by spot welding a thermocouple to the notch and then recording the temperature of the specimen as the sample is removed from the Liquid Nitrogen and tested. Depending on the amount of time the sample was allowed to heat up after being removed from the Liquid Nitrogen, the temperature range from -185C to room temperature could be addressed.

Table II: Summary of solder systems evaluated for this study.

Solder Systems Evaluated	
1	Sn-1.0 wt.%Pb
2	Sn-2.0 wt%Pb
3	Sn-37 wt%Pb
4	Sn-36 wt%Pb 2 wt%Ag
5	Sn-50 wt% Pb
6	Sn- 90 wt%Pb

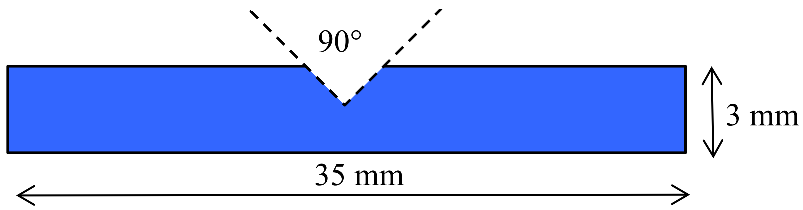

Figure 2: Schematic of modified Charpy test specimens.

3. Results & Discussion

The results of our modified Charpy tests are summarized in Figure 3, focusing on the ductile to brittle transitions in the Pb-based alloys. With the exception of Sn- 90 wt% Pb, all of the Pb-based alloys studied showed an increase in Charpy impact energy with a decrease in temperature, reaching a maximum right before the transition temperature (Figure 3). The sharp transition observed in the Pb-based alloys indicates a transition from ductile behavior to brittle behavior. Sn has a body centered tetragonal (BCT) crystal structure, , while Pb has a face centered cubic (FCC) structure. Figure 4 shows the crystal structure and slip systems associated with the BCT unit cell [9]. The β-Sn can access up to 6 slip systems $\langle 001\rangle\{110\},\{100\}$ as predominant slip systems and possibly $\langle 10\bar{1}\rangle\{101\},\{121\}$, however the stress required to activate these slip systems drastically increases as temperature decreases. In FCC alloys, the availability of multiple close-packed slip systems $\langle 1\bar{1}0\rangle\{111\}$ results in ductile behavior that is relatively independent of temperature [10]. As seen in Figure 3, as more Pb is added to the alloy composition, the alloy behavior more closely resembles a pure FCC alloy, where no transition is observed. In the case of the Sn- 90wt% Pb alloy, the FCC-like behavior is clearly evident even though this is still a two-phase material.

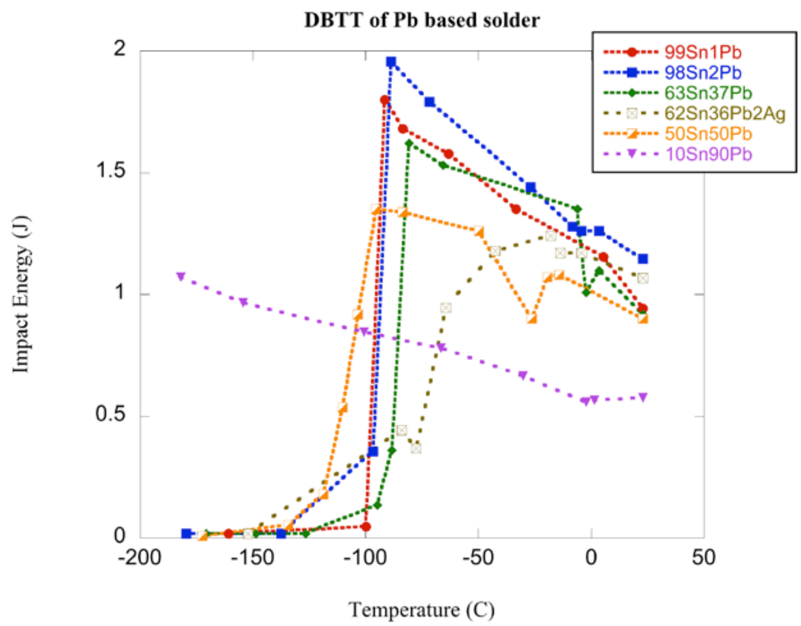

Figure 3: Results from modified Charpy impact tests for Pb-based solders. A clear DBTT can be observed for all but the 10Sn90Pb samples.

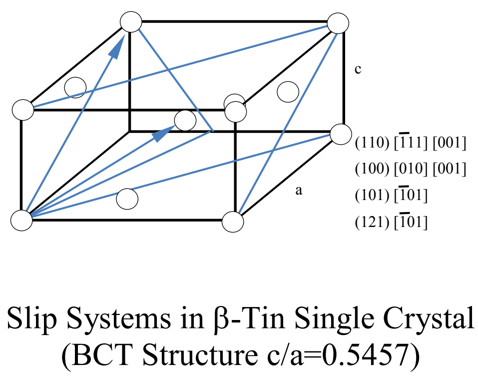

Figure 4: The unit cell of the BCT tin and the common slip systems after[9].

4. Future Work

In addition to the Pb-based alloys that were evaluated for this study, future work will also focus on the Pb-free alloys summarized in Table III below. In particular we will address the specific microstructure of the alloys tested to evaluate crack propagation and whether the rule of mixtures regarding BCT/FCC phase behavior is reflected in the DBTT data and sample fracture behavior.

Table III: Summary of future Pb-free alloys to be characterized.

Solder Systems Evaluated

1	Sn- 3 wt%Cu 1 wt%Ag
2	Sn- 5 wt%Sb
3	Sn- 50 wt%In

5. Summary

In summary, this study evaluated the ductile to brittle transition in six different Pb-based solders. Solder systems that were comprised of predominantly Sn exhibited classic ductile to brittle behavior. The more Pb an alloy contained, the more the behavior trended towards that of a pure FCC material. Future work will focus on the exact origins of this behavior and look at Pb-free solder systems to evaluate trends and mechanisms useful for aerospace alloy design.

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References

[1] Wiley J. Larson & James R. Wertz, Space Mission Analysis and Design, (New York, NY: Springer Press, 1999), pp. 428-434.
[2] A.A. Shapiro, C. Tudryn, D. Schatzel, S. Tseng, "Electronic Packaging Materials for Extreme, Low Temperature, Fatigue Environments." IEEE Transactions on Advanced Packaging, Volume 23, Number 2, May 2010, pp. 408-420.
[3] Vision and Voyages for Planetary Science in the Decade 2013-2022, Committee on the Planetary Science Decadal Survey; National Research Council, (Washington D.C., National Academy Press, 2011), ISBN: 0-309-20955-2.
[4] W. K. Jones, Y.Q. Liu, and M. Shah, "Mechanical Properties of Sn-In and Pb-In Solders at Low Temperature," International Symposium on Advanced Packaging Materials (1997): 64-66.
[5] A. D. Styrkas , "Growth of Gray Tin Crystals", Inorganic Materials, Vol. 39, No. 7, pp. 683–686, 2003
[6] D. Di Maio and C.P. Hunt, "Monitoring the Growth of the α Phase in Tin Alloys by Electrical Resistance Measurements", Journal of Electronic Materials, vol. 38, no. 9, pp.1874-1880, 2009.
[7] A.A. Matvienko and A.A. Sidelnikov, J. Alloys Comp. 252, pp. 172–178 (1997)
[8] C. Zachrisson, H. Kozachkov, S. Roberts, G. Kaltenboeck,R. Conner, M. Demetriou, W. Johnson, D. Hofmann JMR, Vol. 26, No. 10, May 28, 2011.
[9] S.N.G. Chu, J.C.M. Li, Mater Sci. Eng. 39, 1 (1979).
[10] A. Kelly and G.W.Groves, Crystallography and Crystal Defects, (Hendon, VA, TechBooks, 1970), pp. 175-176.