FABRICATION AND PROPERTIES OF A TIN-LITHIUM ALLOY *

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OBJECTIVE

The objectives of this task are to fabricate an Sn-Li alloy containing 25 at.% Li, which is being considered as a potential coolant in a fusion device, and to evaluate the alloy's physical and chemical properties.

SUMMARY

An Sn-25 at.% Li alloy has been fabricated by starting with pure Sn and Li. The alloy was cast into various shapes and sent to researchers in several organizations in the United States and abroad for property measurements. Melting temperature of the alloy is 334°C and its density is 6.36 g/cm^3.

INTRODUCTION

Tin-lithium alloys seem to have several attractive thermo-physical properties, in particular thermal conductivity and heat capacity, when compared with those for another coolant, Pb-17 at.% Li [1]. Furthermore, Sn-Li coolant exhibits low vapor pressure, a significant benefit in the design of the free-flow first wall and divertor that will handle high heat flux and high neutron wall loading in fusion devices [2,3]. Even though conceptually Sn-Li is a potential coolant/breeding material, several technical issues such as physical and chemical properties, materials compatibility, activation characteristics, and breeding potential, must be addressed. We have therefore fabricated an Sn-Li alloy containing 25 at.% Li which will be used by researchers from several institutions in U.S. and abroad to evaluate various properties of the material.

FABRICATION PROCEDURE

Figure 1 is a schematic illustration of the Sn-Li phase diagram, which shows that the melting temperature of the alloy increases monotonically with an increase (above 5 at.%) in Li concentration in the alloy. For tritium breeding, an Li concentration of at least 20 at.% is suggested; a composition of Sn-25 at.% Li was selected for fabrication. The melting temperature for this alloy will be ≈330°C. The phase diagram also shows several Li-Sn compounds (such as Li_4Sn, Li_7Sn_2, Li_5Sn_2, Li_2Sn, and LiSn) that have melting temperatures of 485 to 783°C. If these high-melting compounds form during alloy fabrication, the mixed alloy must be heated to very high temperatures to melt these phases and to develop an alloy with a homogeneous composition. Therefore, it is essential that none of these high-temperature phases form during mixing of Sn and Li to make the alloy. To avoid this, an apparatus was assembled in which Li is gradually added to Sn so that the Li concentration increased monotonically from 0 to 25 at.%, with an associated increase in melting temperature from 250 to

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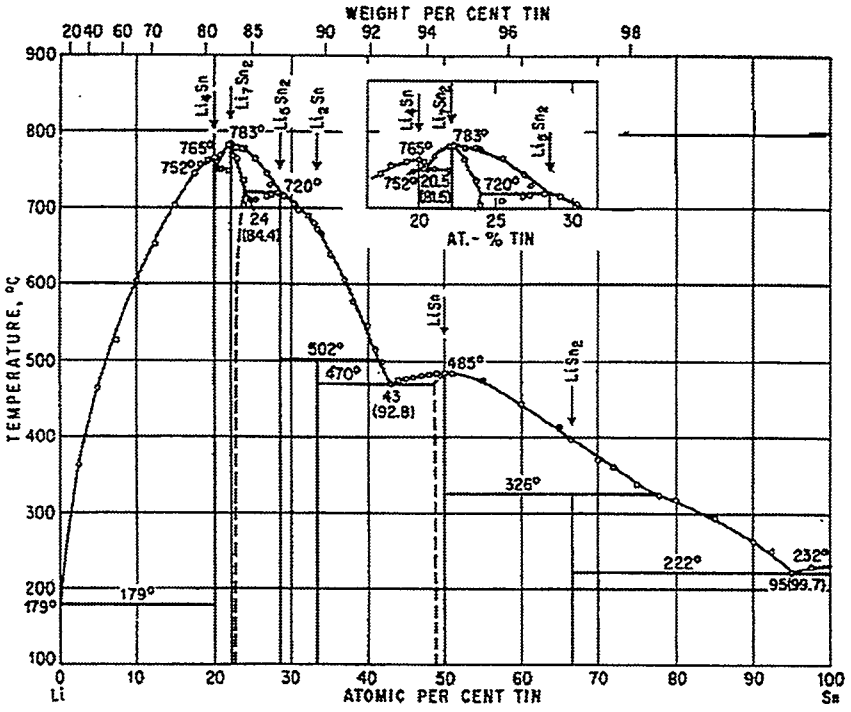

Figure 1. Schematic illustration of Sn-Li phase diagram.

350°C. Furthermore, a stainless steel stirrer was used to stir the liquid while Li was continuously added to Sn to improve the composition homogeneity of the final Sn-Li alloy.

The Sn, obtained from Ames Metal Products, was in the form of 25 x 12.5 x 250 mm castings with a purity of 99.95 wt.% Sn. Lithium was obtained from Cypress Foote Mineral in the form of rods of 12.5 mm dia x 275 mm length. Figure 2 includes a photograph of the Sn ingots and Li contained in an inert environment within a sealed metal can. The equipment used to mix Li into Sn was assembled with components made of austenitic stainless steel. The mixing chamber was a vessel 0.4 m dia. x 0.5 m high and was equipped with a viewing port, thermowell, drain, and two insertion ports at the top. One of these insertion ports was used to assemble a stirrer mechanism and the other was used as a line (12.5-mm-dia. tubing) for transferring Li into Sn vessel. Li was contained in a second vessel (0.15 m dia. X 0.35 m high) above the Sn vessel. Resistance heaters and thermal insulation were applied to both Li and Sn vessels, as well as to the Li transfer line. The gas phase of the two vessels were independently connected to a source of vacuum and purified argon gas. Figure 3 shows photographs of the experimental assembly in different stages of construction.

Calculations on the requirements for Sn and Li were made to produce ≈15 L of the Sn-25 at.% Li alloy. Using the density values of 0.54 and 7.3 g/cm^3 for pure Li and Sn, respectively, we calculated the density of Sn-25 at.% Li alloy as 6.36 g/cm^3. To produce 15 L of the Sn-Li alloy, the initial amounts of Sn and Li needed were 93.5 and 1.81 kg of Sn and Li, respectively.

Figure 2. Photographs of starting materials: Sn (left) and Li (right).

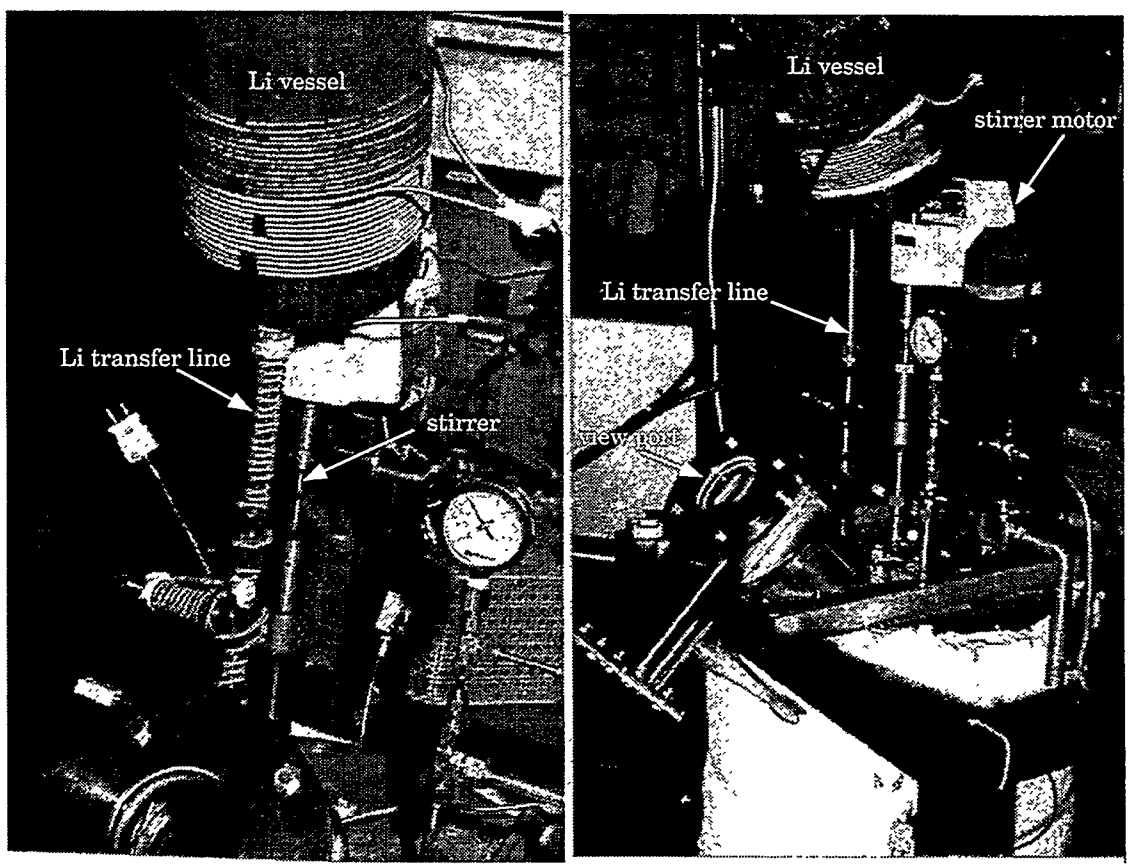

Figure 3. Photographs of experimental set up at different stages of assembly. The key parts of the assembly are Sn vessel (with insulation), Li vessel at the top, Li transfer line, Stirrer with a motor, and a view port.

The initial step was to melt the slender bars of pure Sn in the lower vessel. Since there were a large number of bars, as many as possible were inserted into the vessel through the view port and the system was then closed, evacuated, and back-filled with Ar. The evacuation/refill operations were repeated three times prior to heating the vessel containing Sn. The bars were then melted and cooled. Two loadings of Sn and melting were required to obtain the required 93.5 kg of pure Sn in the vessel. The smaller Li vessel also required two loadings to obtain 1.81 kg of Li. Rods of Li were removed from their secondary protective plastic containment in the vicinity of the Li vessel and inserted in to the Li vessel with minimal exposure to ambient air. In addition, flowing Ar from the vessel mitigated the potential for unwanted oxygen contamination during the transfer. At the end of each loading, the Li was melted and slowly added to the molten Sn in the mixing chamber. An attempt was made to video tape the first addition of Li to Sn, but the viewing window did not allow a clear view of the stream of Li striking the molten Sn, the lighting was poor, and after the initial exothermic reaction, the window became clouded. The results were less than desirable but a VHS tape of the operation does exist.

In the first mixing operation, Sn and Li vessels were maintained at 240 and 200°C, respectively. Upon adding Li to Sn, the temperature of the mixing chamber increased to 419°C. The second batch of Li was added to the mixing chamber maintained at 320°C. The temperature of the mixing chamber rose to 512°C after addition of the second batch of Li. The Sn-Li alloy was maintained at this temperature for 15 min while the stirrer was operating at high speed. A sample of the alloy was taken by casting it under vacuum in a heated stainless steel tube (≈0.5 m long x 12.5 mm dia.) attached to the mixer vessel drain. The initial analysis indicated an Li content of 0.83 wt.%, which was significantly lower than the additions made to the vessel, probably because of the inhomogeneity of the melt. The mixing was held at 400-500°C with vigorous stirring approximately eight times a day for three days to dissolve any high-temperature compounds and to homogenize the melt. A second sample taken in the same manner as before showed an Li content of 1.96 wt.%, based on an analysis of duplicate samples. This value corresponds to an alloy composition of Sn-25 at.% Li. Table 1 lists the concentration of various substitutional and interstitial elements in the alloy.

Figure 4 shows castings of several shapes and sizes made with a copper mold. Material from various product forms have been sent to CEA-CEREM/SCECF in France, the University of Tokyo in Japan, UCSD Fusion Energy Research in San Diego, and the University of Illinois at Urbana-Champaign for measurement of various physical properties. Figure 5 shows scanning electron photomicrographs of the alloys in two magnifications. The two-phase solidified structure consists of Sn and $SnLi_2$ at room temperature. Vickers hardness measurements indicated the average hardness values for the light and dark phases to be ≈20 and 85, respectively. Figure 6 shows the temperature/time cooling curve generated for the alloy. The point where the slope of the line changes (334°C) indicates the melting temperature for the alloy. This agrees well with the temperature depicted in the phase diagram (see Fig. 1) for an alloy with a composition of Sn-25 at.% Li.

Table 1. Chemical composition of Sn-Li alloy fabricated in current project

Element	Concentration (wt.%)	Element	Concentration (wt.%)
Ag	0.017	Na	<0.001
Al	<0.01	Ni	0.02
Ba	0.002	Pb	0.02
Be	<0.001	Si	<0.05
Ca	<0.001	Sr	<0.001
Cd	<0.001	Ti	<0.005
Co	<0.01	V	<0.005
Cr	0.03	Zn	<0.001
Cu	<0.005	Zr	<0.005
Fe	0.08	O	0.091
K	0.011	N	<0.0002
Li	1.96	C	0.085
Mg	<0.001	S	<0.0035
Mn	0.017	Sn	balance

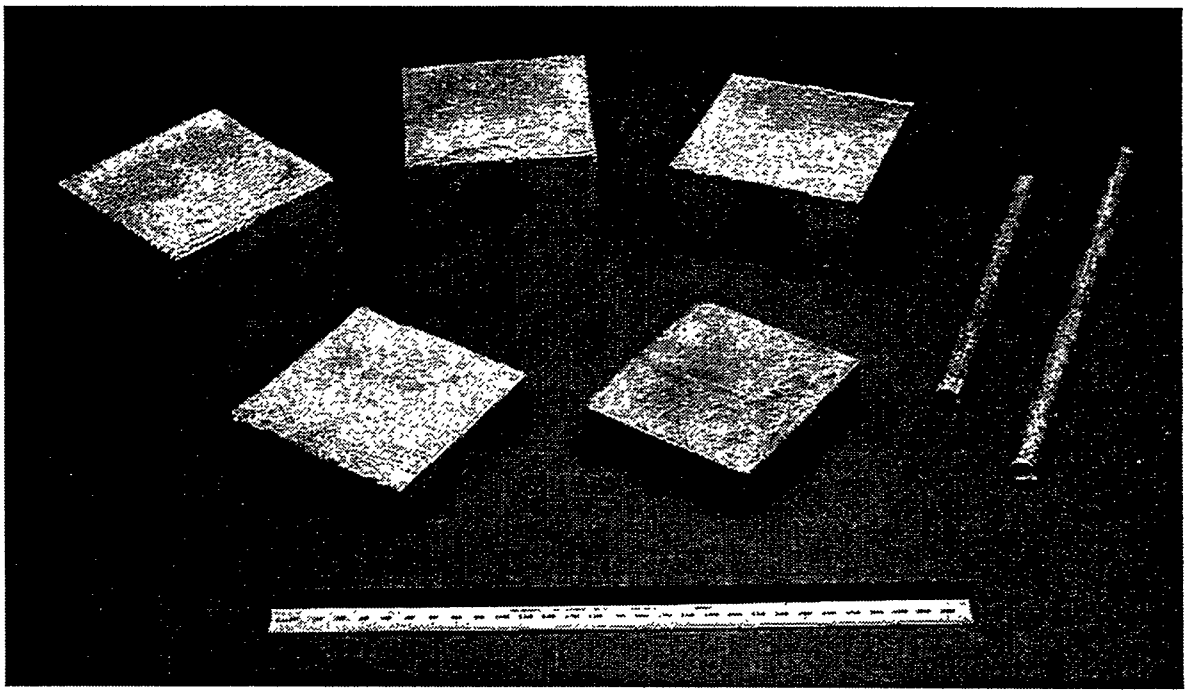

Figure 4. A photograph of different shapes of an Sn-25 at.% Li alloy fabricated in the program

REFERENCES

1. M. A. Futterer, G. Aiello, F. Barbier, L. Giancarli, Y. Poitevin, P. Sardain, A. Li Puma, G. Ruvutuso, and G. Vella, "On the use of tin-lithium alloys as breeder material for blankets of

fusion power plants," presented at 9th Intl. Conf. on Fusion Reactor Materials, Oct. 9-15, 1999, Colorado Springs.

2. D. K. Sze, "The development of Sn-Li coolant/breeding material for APEX/ALPS applications," presented at Intl. Symp. on Fusion Nuclear Technology, Rome, Sept.1999.

3. D. K. Sze, R. F. Mattas, Z. Wang, E. T. Cheng, M. Sawan, S. J. Zinkle, and K. A. McCarthy, "Sn-Li, a new coolant/breeding material for fusion applications," presented at 9th Intl. Conf. on Fusion Reactor Materials, Oct. 9-15, 1999, Colorado Springs.

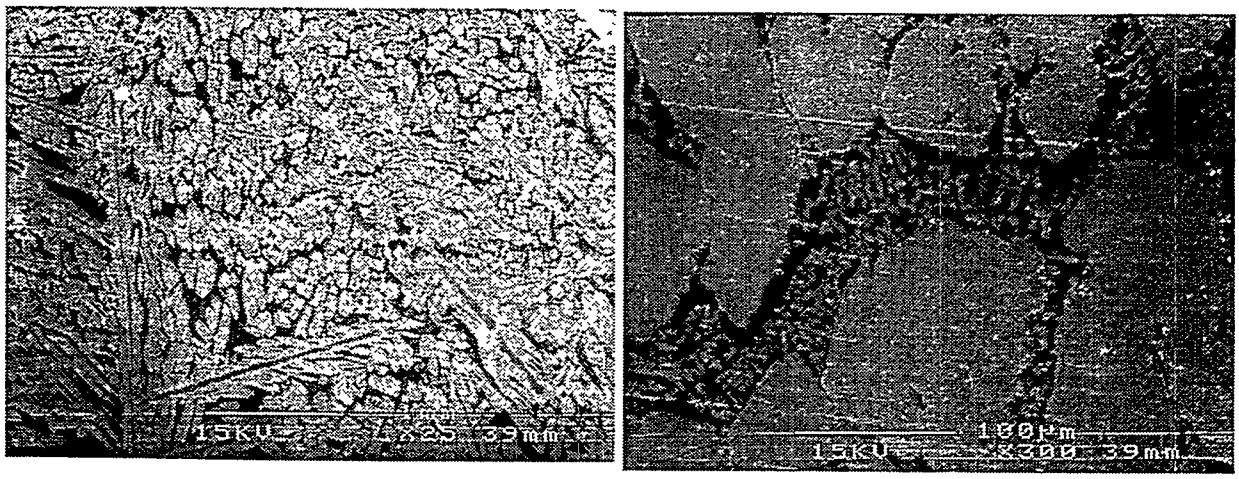

Figure 5. SEM photomicrographs of an Sn-25 at.% Li alloy fabricated at Argonne National Laboratory

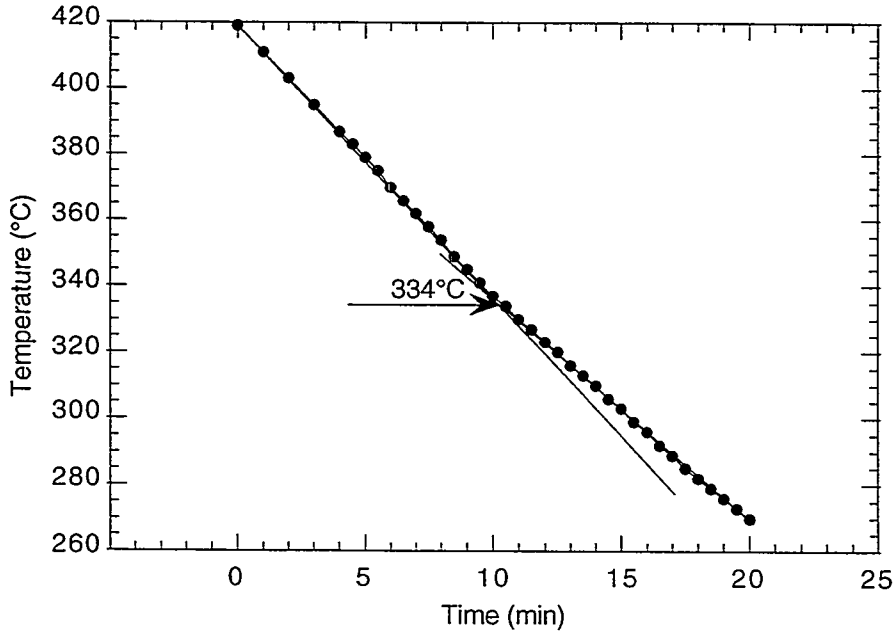

Figure 6. Temperature/time cooling curve for an Sn-25 at.%Li indicating a melting temperature of 334°C.