

additives), preferably in the form of a master alloy, to a melt of pure lead at a temperature above the melting point of lead. Care should be preferably taken to prevent excessive oxidation during manufacture and use, although under most practices this is not a problem when a master alloy of Pb with less than 0.1% of the alkali metal is used to prepare the final alloy.

In an advantageous embodiment, a high lead solder composition comprises Li in an amount of about 0.001–0.02%, Te as a grain-size controlling additive in an amount of about 0.005–0.1%, and lead comprising the remainder of the composition. In another advantageous embodiment, a high lead solder composition comprises Li in an amount of about 0.001–0.02%, Mg as a grain-size controlling additive in an amount of about 0.005–0.05%, and lead comprising the remainder of the composition. In yet another advantageous embodiment, a high lead solder composition comprises Li in an amount of about 0.001–0.02%, Te and Mg as grain-size controlling additives in amounts of about 0.005–0.1% and 0.005–0.05%, respectively, and lead comprising the remainder of the composition.

#### EXAMPLE 1

A small sample of several solder alloys in accordance with the invention were reflowed above the liquidus point of the alloy, then solidified onto copper substrates. The samples were then put into a furnace at 205° C. for 200 hours to simulate exposure to a harsh environment during use. The samples were sectioned, polished, etched and examined metallographically. The grain size data are shown in Table 1. It is seen that the presence of Te, Se, Ce, and Mg contribute to a refined grain size in the presence of Li-containing Pb alloy. These alloys meet the target grain size of less than 200  $\mu\text{m}$  after 200 hours of exposure at 205° C., which is advantageous, since small grain size correlates with improved fatigue performance.

TABLE 1

Grain size 205° C. ( $\mu\text{m}$ )	Pb	Li	GCA <sup>1</sup>
100 × 100	99.99	0.0072	
50 × 50	99.98	0.0027	0.01 Te
50 × 50	99.98	0.0030	0.01 Se
50 × 50	99.95	0.0010	0.04 Ce
25 × 25	99.89	0.0005	0.01 Mg

<sup>1</sup>Grain controlling additive

A standard wetting balance test was performed on several alloys on a MULTICORE MUST II instrument to determine wetting behavior. The results of this testing are shown in Table 2. The force was measured at 2 seconds and 5 seconds into the test. A large positive number indicates the best wetting. For manufacturability a positive force is desired, since this indicates rapid and adequate wetting for assembly of components on circuit boards. It is seen that a positive value was obtained for each of the alloys of the present invention at 5 seconds, i.e., those containing Li and/or GCAs, and that the alloys containing Te and Mg had good performance at 2 seconds as well. Note also that none of the alloys in accordance with this disclosure showed “dewetting” behavior (shown by an decrease in F between 2 and 5 sec.), while the prior art compositions P1 and P2 evidenced undesirable dewetting behavior.

TABLE 2

Wetting Balance						
	F(2s) (mN)	F(5s) (mN)	Pb	Ag	In	Li Other
	0.16	0.20	99.98			0.0027 0.01 Te
	-0.06	0.09	99.98			0.0030 0.01 Se
	-0.02	0.10	99.95			0.0010 0.04 Ce
	0.04	0.10	99.89			0.0005 0.01 Mg
P1	0.16	0.13	92.5	2.5	5	
P2	0.39	0.35	8.8	10	2	

#### EXAMPLE 2

The effect of the addition of lithium to a high lead alloy is illustrated in the following example, showing the wetting behavior of a high-lead alloy on a polished copper substrate. An OFHC (oxygen free high copper) polished copper substrate, cleaned with sulfuric acid for approximately 30 seconds, followed by rinsing and methanol washing, was used as the substrate. Flux was applied to the front and the back of the substrate. A pellet of solder was placed on the copper substrate, and additional flux was placed on top of the pellet. The substrate was then placed on a programmable hot plate surface at a temperature of 340° C., then removed 20 seconds after the melting of the alloy. The height and diameter of the melted pellet was measured, and the contact angle determined. As can be seen from the data in Table 3, the contact angle for the lead alloys containing a very small amount, i.e., 0.012%, of lithium is greatly improved compared to pure lead, and is comparable to the good wetting performance of the 90/10 lead/tin alloy. Furthermore, it can be seen that addition of an amount of lithium greater than 0.01% results in an decrease of wetting ability, surprisingly showing that only very small amounts of lithium added to a substantially pure lead alloy impart greatly improved wetting properties to the alloy.

TABLE 3

	Diameter (mm)	Height (mm)	Contact Angle (degrees)
90% Pb/10% Sn	3.58	0.365	23.1
	-3.77	0.440	26.3
99.82% Pb/0.12% Li	3.10	0.580	41.0
	3.22	0.515	35.5
99.988% Pb/0.012% Li	3.31	0.425	28.8
	3.76	0.430	25.8
100% Pb	2.60	0.635	52.1
	2.78	0.630	48.8
	3.15	0.638	44.1

The foregoing description is meant to be illustrative of the invention. Other embodiments and variations will be apparent to those of ordinary skill in the art without departing from the inventive concepts contained herein. Accordingly, this invention is to be viewed as embracing each and every novel feature and novel combination of features present in or possessed by the invention disclosed herein and is to be viewed as limited solely by the scope and spirit of the appended claims.

What is claimed is:

1. A high lead solder composition for high temperature electrical applications consisting of:

- about 0.0005–0.1 wt %, based on the total weight of said solder composition, of an alkali metal selected from the group consisting of Na, K and Li;
- a grain-size controlling additive selected from the group consisting of Ce, Ba, Pr, Nd, Sm, La, Y, Eu, Gd,