the field line can sustain) most of the time. As a result, the electrons contribute most of the heating at 0-deg inclination. Magnetic storms are not important in this orbit because they do not cause major increases in the electron flux (see Fig. 1). Also, at this altitude, the offset dipole of the Earth's magnetic field does not produce any significant effects. However, the distortion of the magnetosphere due to the solar wind does produce local-time variations. These local-time effects are significant in the energetic electron flux, especially in the >500 keV range, and produce the difference between average and peak heating at 0 deg plus much of the variation seen in the peak heating as a function of inclination.

At geosynchronous orbit, the peak heating in high inclination orbits is due to protons even though the average heating is primarily due to electrons. This is caused by hot plasma conditions beyond $L = 6.6$. In this high-altitude regime, there are usually large fluxes of protons with energy in the tens of keV range, while few energetic electrons are present.

Summary

The geomagnetically trapped-particle population can produce significant heating in some systems. Model calculations indicate that greater than 0.5 W/m^2 instantaneous heat input may be encountered. For some orbits, geomagnetic storms can produce an increase in the energetic particle flux that results in a further increase in the average heat input by half an order of magnitude and an increase of an order of magnitude in the peak heat input. In general, low-altitude orbits are benign and orbits that pass through the heart of the outer zone (the equator at $L = 4$) are fairly severe. The geosynchronous orbit is between these extremes. Also, the higher the inclination of the orbit, the lower the average heating.

Acknowledgments

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Effects of Crucible Wetting During Solidification of Immiscible Pb-Zn Alloys

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Introduction

AN alloy containing a liquid-phase miscibility gap has a two-phase field consisting of immiscible liquids. The Pb-Zn (lead-zinc) system has such a liquid-phase miscibility gap.[1,2] Hundreds of immiscible alloys exist.[3] However, the processing of these alloys is generally considered impractical because of their immiscibility in the liquid state. In the immiscible field the two liquids separate due to their different densities, similar to the way oil and water separate.

The low-*g* environment of space provides a unique opportunity to process these alloys. In low *g*, it is possible that the immiscible liquid droplets that form in the parent liquid will not segregate since gravitational Stokes settling forces have been nearly eliminated. The ability to study this large group of alloys without gravity-induced segregation has sparked new interest in these alloys and their possible applications. Liquid-phase miscibility gap alloys are presently being developed for electrical contact applications. Other possible uses are as superconductors, catalysts, permanent magnets, bearings, and superplastic materials.[4,5] To develop immiscible alloys for these applications, ingots are needed with a uniform distribution of phases. Solidification of these alloys in low *g* will help us to attain this goal and contribute to our understanding of the processes by which immiscible alloys segregate on Earth.

This research was done in support of the design and development of a planned Space Shuttle experiment using a getaway-special canister.[6,7]

Many of the immiscible alloys previously processed in low *g* have resulted in severely segregated structures due to unexpected fluid flow and other uncertain experimental parameters.[8-12] These experiments have shown the importance of the wetting behavior of the two immiscible liquids and the crucible. Upon cooling into the immiscible phase field, small droplets of at least one of the two liquids form. The two liquids have different compositions and a different equilibrium wetting angle with the crucible. Consider, for example, a Pb-rich droplet in a melt of average composition Zn-40 wt % Pb just touching the alumina crucible containing the melt. If the Pb-rich alloy preferentially wets the alumina relative to the parent liquid, the Pb-rich droplet will tend to spread out on the crucible. This motion will cause fluid flow, thus helping other Pb-rich droplets to contact the crucible. This autocatalytic process promotes further coalescence and segregation of the two liquids. In low-*g*, this may result in the Pb-rich liquid coating the inside of the crucible and the Zn-rich liquid collecting in the center. Thus, nearly complete segregation of the two phases may result even though gravity-driven sedimentation has been eliminated.

Macrosegregation due to preferential wetting of this type may be avoided in less concentrated alloys ($\simeq 10$ vol % or less) by the majority phase preferentially wetting the crucible.[13,14] This of course does not help us when attempting to process more concentrated alloys. Also, there is evidence that minority-phase droplets that do not wet the crucible may be pushed away from the crucible wall causing flow and macrosegregation.[14] Thus, a knowledge of the wetting behavior is required for the interpretation of structures and the efficient design of crucibles for low-*g* experimentation.

Cahn's theory states that complete preferential wetting of the crucible by one of the liquids occurs at temperatures near the critical point at the top of the immiscible dome.[15,16] The balance of interfacial energies between the two liquids and the crucible is given by

$$\gamma_{L_1 L_2} \cos\theta = \gamma_{SL_1} \gamma_{SL_2} \tag{1}$$

where the angle θ is the dihedral angle shown in Fig. 1, $\gamma_{L_1 L_2}$ is the interfacial free energy at the boundary between the two immiscible liquids, and γ_{SL_1} and γ_{SL_2} are the solid-L_1 and solid-L_2 interfacial free energies, respectively. Complete wetting occurs when $\theta = 0$ and at undefined values of $\cos\theta$ such that

$$\gamma_{L_1 L_2} < |\gamma_{SL_1} - \gamma_{SL_2}| \tag{2}$$

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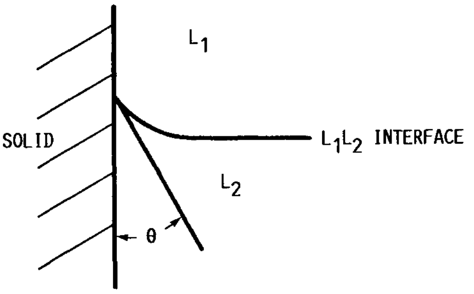


Fig. 1 Dihedral angle θ at three-phase junction measured through preferential wetting liquid.

Heady and Cahn[16] showed in the methylcyclohexane-perfluoromethyl-cyclohexane immiscible system that the left-hand side of Eq. (2) decreases to zero with a $\Delta T_{uc}{}^{1.3}$ relationship and the right-hand side as $\Delta T_{uc}{}^{0.3}$, where ΔT_{uc} is the undercooling or temperature difference below the immiscible dome. Thus, at some temperature below the critical temperature, perfect wetting of any solid by one of the liquid phases is expected. By choosing the crucible for a particular alloy so that the difference in suface energy between the solid and two liquid phases is minimized, the effects of preferential wetting may be diminished and possibly avoided.

Many theories have been developed in an effort to model and predict solidification and the final structure of immiscible alloys.[12,17] Attempts have been made to study and model separation and coarsening mechanisms.[15,18] These mechanisms are often overshadowed by the effects of preferential wetting, thus making their study difficult if not impossible. Elimination of crucible wetting processes would enable us to study these mechanisms as well as help in the production of a less-segregated product in low g. The objective of the present work was to identify a crucible material in which the fluid flow induced by the wetting behavior between the Pb-Zn alloy and crucible would be eliminated or significantly reduced.

A series of experiments was conducted and has qualitatively shown the competitive wetting characteristics between the Pb-Zn immiscible alloy system and various crucible materials. Approximate wetting angles at the crucible-Pb-Zn interface were measured in an effort to find a crucible such that the interfacial energies between the crucible and liquids is minimized. It is hoped that this analysis will assist in the choice of crucibles, for other alloys as well as Pb-Zn, which will reduce the fluid flow and segregation normally caused by competitive wetting in low g.

Experimental Procedure

The crucible materials listed in Table 1 were cut to fit inside 5/8 in. i.d. fused-silica test tubes and placed vertically in the tube with approximately 25 g of Pb-35 wt % Zn (when solid approximately Pb-46 vol % Zn). The Pb and Zn material used was 99.999% pure. The crucible material and alloy were sealed in a fused-silica tube or a stainless steel container. In some cases, as noted in Table 1, crucibles were made of the test material. In these cases, wetting angles were measured from the container crucible itself. Figure 2 shows schematically the test tube and crucible test material arrangement. All samples were heated above 816°C, briefly removed from the furnace and mixed by gentle agitation, reheated to above 816°C, soaked at temperature for at least 10 min, and slowly cooled. The average cooling rate through the immiscible liquid region and solidification was 0.2°C/s.

After solidification ingots were sectioned longitudinally along the centerline, perpendicular to the crucible test material, and mounted in metallographic epoxy such that the cross section could be viewed. Micrographs were taken after polishing and approximate wetting angles measured from the micrographs. The wetting angles listed in the table are meant only as a qualitative comparison of wetting behavior of the different crucible materials. The wetting angle θ was always measured through the preferential wetting liquid as shown in Fig. 1.

Table 1 Preferential wetting liquid and qualitative wetting angles between Pb-Zn immiscible liquids on various crucible material

Crucible material	Form of material	Preferential wetting liquid	Postsolidification wetting angle θ, deg
Niobium	Sheet	Zn	0
Cobalt	Plate	Zn	0
Nickel	Plate	Zn	—
SiC	HIPed bar	Pb	8
Macor glass	Crucible	Pb	9
Fused silica	Crucible	Pb	11
Al_2O_3	HIPed bar	Pb	11
ZYP coating "S-prime-mod"	Crucible	Pb	13
Boron nitride	Plate	Pb	14
Molybdenum	Sheet	Pb	24
Tantallum	Sheet	Pb	27
Tungsten	Sheet	Pb	37
Carbon	Plate	Pb	48

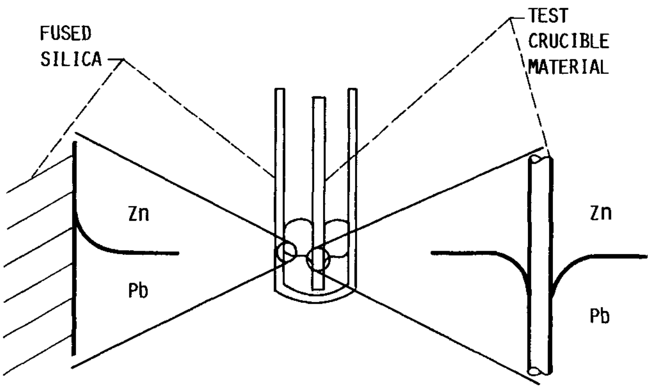
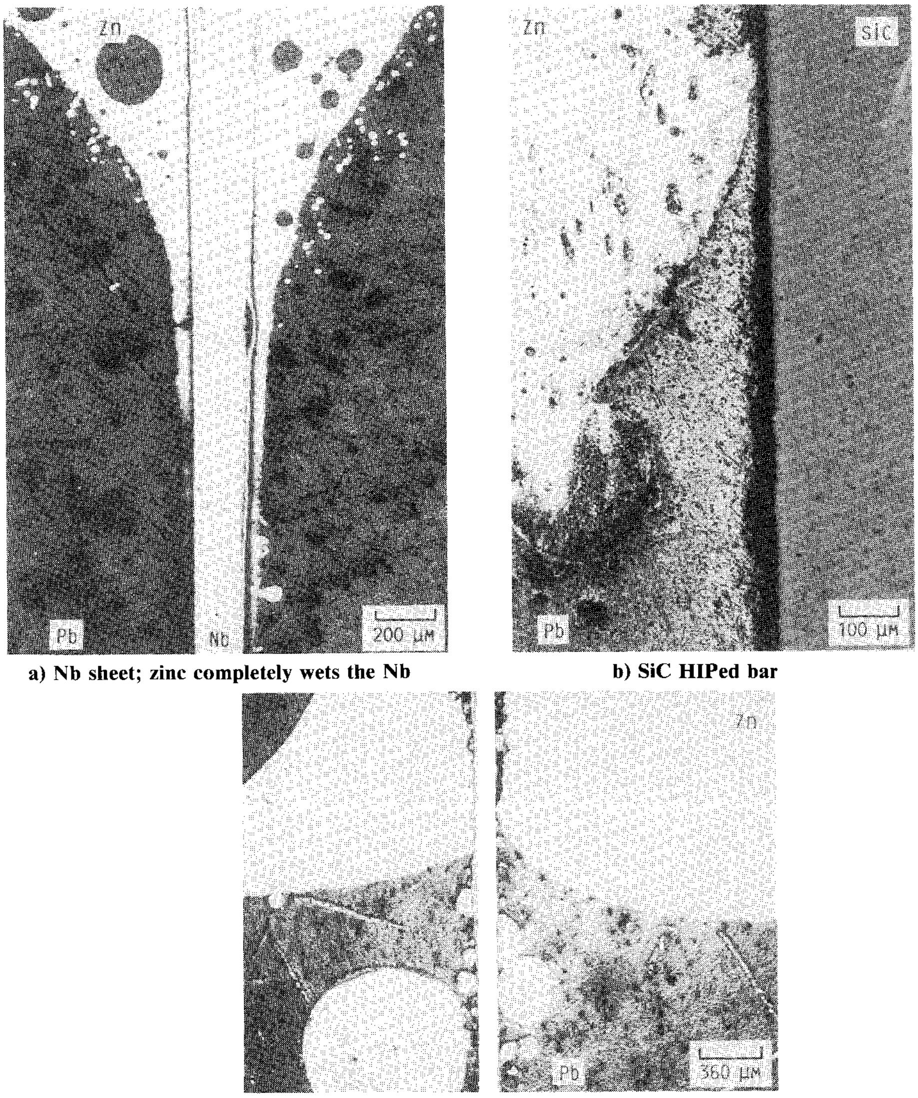


Fig. 2 Schematic cross section of silica test tube, Pb–Zn ingot, and test crucible material. (Left: wetting at container crucible wall; right: wetting at test crucible material.)

Results

Table 1 shows the crucible material tested, preferential wetting liquid phase, and approximate wetting angle. The material Macor is a fully dense machinable glass supplied by Corning Glass. The ZYP coating was used on a stainless steel container and is a silica-based paintable coating designed for metal substrates and supplied by ZYP Coatings Inc. The SiC was a hot-isostatically-pressed rectangular bar. Each wetting angle in Table 1 is the mean of several measurements. Three to ten measurements were made to determine θ. The higher number of samples were made of materials in which large scatter appeared in θ. Figures 3a–3c show Pb-Zn wetting behavior on Nb, SiC, and W.

All of the materials preferentially wet by Zn were attacked by the Zn and reacted with it. Niobium reacted the least and nickel the most. Nickel completely dissolved in the molten zinc. The reaction with niobium was slightly less severe than that with cobalt. Zinc completely wet the cobalt (Co) plate and reacted to form Co-Zn intermetallics at the cobalt plate-Zn interface. Such potential reactions must be considered when choosing crucible materials. If Zn wetting is required and contamination of the Zn can be tolerated, Nb is the recommended crucible material.



a) Nb sheet; zinc completely wets the Nb

b) SiC HIPed bar

c) W sheet

Fig. 3 Optical photomicrograph of Pb-Zn ingots with various crucible test materials placed down the center prior to melting.

Of the crucible materials that were preferentially wet by Pb, carbon (C) and tungsten (W) had the two largest three-phase dihedral angles, θ. Therefore, C and W are the best choice of the crucible materials tested here when preferential wetting by Pb over Zn and a minimization of $(\gamma_{SL_1} - \gamma_{SL_2})$ is desired.

Summary

To minimize the fluid flow and segregation caused by preferential wetting of one of the immiscible liquid phases in low g, the majority phase should preferentially wet the container and the surface energy difference $(\gamma_{SL_1} - \gamma_{SL_2})$ minimized. Smaller surface-energy differences are characterized by larger values of the dihedral angle θ.

Thirteen readily available materials were investigated as possible container materials for Pb-Zn alloys. The phase that preferentially wet each test material was determined and approximate contact wetting angles measured.

The three crucible materials preferentially wet by Zn were also chemically attacked by the Zn. Niobium was the least reactive and is the recommended material for use when preferential wetting by Zn is required and Nb contamination of the Zn can be tolerated.

Of the crucible materials preferentially wet by Pb, C and W had the two largest dihedral angles and thus are the recommended materials when wetting by Pb over Zn is required and minimization of fluid flow due to critical and preferential wetting is desired.

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