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DEFORMATION OF SEMI-SOLID METALS - REFINING, STRENGTHENING, AND RHEOLOGICAL BEHAVIOR

FINAL REPORT

Merton C. Flemings

July 15, 1982

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This report covers research activities in the broad area of deformation of solid metals and include work on metal refining, metal strengthening, and rheological behavior. A new process for metal refining is described that combines squeezing the liquid from semi-solid metal with heating of the semi-solid mass. Another new process is described for strengthening metals by squeezing out small quantities of interdendritic material. Fundamental rheological work on the behavior of semi-solid alloys is also described.

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DEFORMATION OF SEMI-SOLID METALS - REFINING, STRENGTHENING, AND RHEOLOGICAL BEHAVIOR

Foreword

This final report covers research activities conducted under ARO sponsorship for the period 16 May 1977 - 15 November 1981. The research activities are all in the broad area of deformation of so`id metals and include work on metal refining, metal strenghthening, and rheological behavior.

A new process for metal refining is described that combines squeezing the liquid from semi-solid metal with heating of the semi-solid mass. The result is a process of extremely high theoretical efficiency. A patent for this process has been applied for.

Another new process is described for strengthening metals by squeezing out small quantities of interdendritic material. A patent has been granted for this process.

Fundamental rheological work on the behavior of semi-solid alloys is also described. This work extends earlier studies on "Rheocasting" and has broad, practical implications. The original Rheocast work, sponsored by ARO, led to a number of subsequent research activities at MIT, and these plus the ARO work led to 27 papers and 11 patents; they are listed in the Appendix. Rheocasting has now finally become a commercial reality. The broad area of forming and otherwise processing of metals in the semi-solid state is one that will assume increasing industrial importance and that will merit future fundamental rheological studies.

Summary of Problem Studied

The overall research has been concerned with fundamental and engineering problems relating to the deformation of semi-solid metals. The essential problems studied in the work were:

1. Purification of semi-solid metals by isothermal deformation ("isothermal refining").
2. Purification of metals by deformation during heating in the semi-solid state ("purification by fractional melting").
3. Rheological behavior of semi-solid metal alloys.

Summary of Results

Initial work in this program was graduate thesis work by Dr. Andrew Lux, who showed that isothermal compression of a semi-solid (naturally molten) metal over a filter resulted in removal of most of the liquid. The work comprised an analytical and experimental study of this process, using a Sn-Pb alloy as the model system. From an engineering point of view, the work showed that refining by the application of pressure is much more efficient than are existing commercial processes which refine by gravity draining of the liquid (e.g., as in the case of refining of high purity aluminum).

The analytical expression derived by Lux and Flemings for "Refining Effectiveness" in isothermal compression is:

$$\frac{\bar{C}_c}{C_0} = \frac{1}{f_c}\{1-[1-f_c(1-W)]^k[\frac{1-f_c}{1-f_c(1-W)}]\} \qquad (1)$$

where $\bar{C}_c$ is final average solid composition (after squeezing), C_0 is original alloy composition, f_c is fraction of starting material that is recovered as purified "cake", W is cake "wetness" (fraction liquid left in the solid cake after squeezing), and k is the partition ratio. The "Refining Effectiveness", $\bar{C}_C/C_0$, is the ratio of final to initial compositions. In fractional solidification this ratio approaches the partition ratio, k, at the limit of zero recovered material (f_c=0).

Lux next showed that refining efficiency could be improved by an astonishing amount if heating and squeezing are performed simultaneously. The expression for refining effectiveness derived in his work is:

$$\frac{\bar{C}_c}{C_0} = \frac{b}{k} f_c^{\frac{1-b}{b}} \qquad (2)$$

where b = k(1 - W) + W. For most systems, the refining effectiveness by the process of equation (2) is orders of magnitude better than that of equation (1) for values of f_c of practical interest. Moreover, the theoretical limit of "refining efficiency" is not k, but is zero at low values of f_c. The validity of this equation was demonstrated experimentally by using a simple Sn-Pb alloy as a model system. It remains an important question for applied research to determine the extent to which this process can be scaled up to commercial application. Certainly, one difficulty is that as the metal becomes increasingly pure, it is necessary to maintain increasing temperature uniformity. A patent for this process is pending.

The Lux work was followed by work of Goodwin, who applied the process to commercial type aluminum alloys to remove a small amount of interdendritic liquid (and associated impurities and segregated phases). He showed this process could be used to produce material of exceptionally high mechanical properties. In this work, 7000 series aluminum alloys were produced with room temperature tensile properties equivalent to or exceeding the best produced by any other process, including consolidation of splat-cooled powders, Figure 1.

During the Goodwin work, we made the observation that in order to squeeze the liquid out of the semi-solid dendritic alloy, it was not necessary (as we had previously thought) to use a fine mesh filter. Instead, we observed that the "filter" could be made up of quite large holes - as large as 1/16-inch across, or even larger. The interdendritic liquid would readily flow through such holes, but the dendrites would clog and not pass. This observation was one factor leading us to want to take a closer, more fundamental look at the deformation behavior of semi-solid dendritic alloys. We thought, on the one hand, that we might find entirely new ways to remove liquid from solid without any filtering approach whatsoever. At the other extreme, we wondered if we could find ways to deform semi-solid dendritic materials without getting segregation or cracking and thereby have a commercial method, other than Rheocasting, for forming metals in their semi-solid state. And we wondered as well if we might find some way, through deformation in the semi-solid state, to break up the dendritic structure so that we could produce an essentially nondendritic "Rheocast" metal that then would deform readily.

Our first study in this area was carried out in the laboratory by M. Suery and co-workers, who studied the behavior of semi-solid Sn-Pb alloys using a parallel-plate viscometer type apparatus.

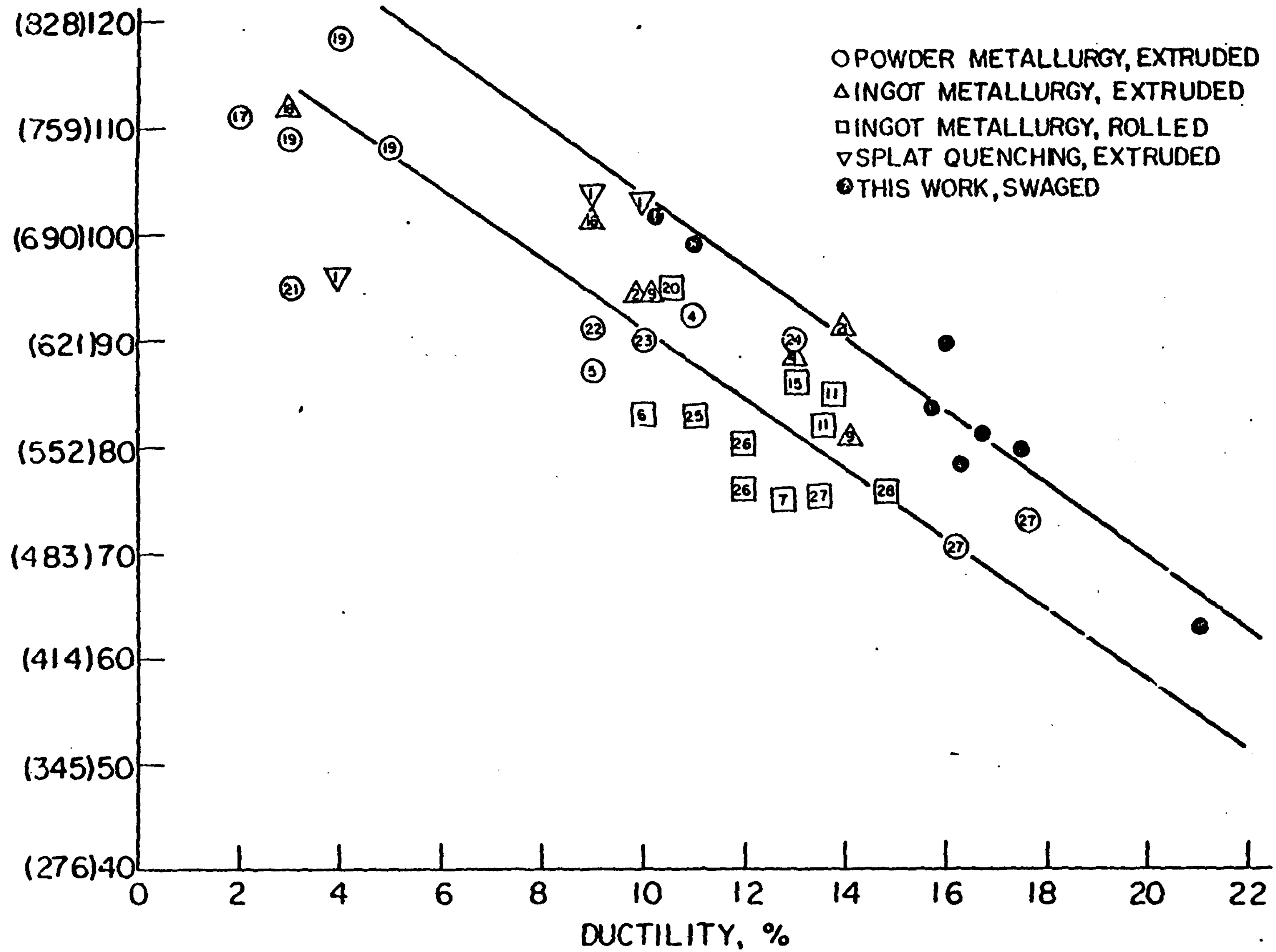

Figure 1. Tensile strength versus ductility for "SFM" 7000 series aluminum alloys, compared with best properties in the literature. Numbers on points refer to source of the data as listed in Goodwin et al.

Small dendritic samples were deformed at constant strain rates ranging from 1.3 x 10^{-3} sec^{-1} to 1.2 x 10^{3} sec^{-1} in the semi-solid state at a temperature just above the eutectic. At the lower rates of deformation, breakdown of the dendrite structure occurred (at strains of 0.2 to 0.4) and a high degree of segregation of the liquid phase was found. For higher rates, the segregation no longer occurred to such a great extent and the alloy deformed more homogeneously. Some related experiments involving compression over a filter were conducted to obtain stress-strain relations in bulk compression for later analysis. It was concluded from this work that compression of alloys in the semi-solid state may be used as a refining process (without a filter) in the low strain-rate range where segregation of the liquid is large. It may also prove useful in the high strain-rate range as a forming method.

The next major activity in this program was carried out by D.A. Pinsky, working with P.O. Charreyron, who continued the Suery work on deformation of semi-solid dendritic alloys. The Pinsky-Charreyron work made two major additional contributions which were:

1. The significant effect of die fraction on deformation behavior and structure in deformation of semi-solid alloys, and
2. The description by a single power law equation of a given semi-solid alloy under a wide variety of different deformation conditions.

The experimental portion of this work was also carried out on semi-solid dendritic Sn-Pb alloys at one degree above the eutectic. Small, cylindrical samples were deformed at an initial strain-rate of 1.3 x 10^{-2} s^{-1} in a parallel-plate apparatus. The friction between the sample and the plates was found to strongly affect both the strength of the material in compresion and the resultant liquid-solid segregation. For low friction, a maximum stress occurred at strains of about 0.3. Above this strain, large cracks were observed. High friction resulted in a much higher degree of segregation than observed for low friction. No maximum stress and no cracking was observed, even for strains as large as 1.2 for high friction.

In separate experiments, cylindrical samples were extruded through cylindrical dies at constant piston velocities ranging from 8.5×10^{-5} m-s^{-1} to 8.5×10^{-5} m-s^{-1} and with reductions of area ranging from 2:1 to 8:1. The deformation occurred in two distinct modes. First, a "compaction" mc during which liquid was expelled and the solid compacted but did not flo through the die, under increasing stress. Secondly, a "flow" mode, during which compacted solid flowed through the die under a constant stress, σ_{extr}.

Experiments involving compression over a filter and compression between parallel plates of alloys of different compositions were performed to examine the effects of the fraction liquid on the rheology of semi-solid dendritic alloys. Correlation and analysis of all experimental results showed the deformation of semi-solid Sn-15% Pb at 184°C to obey the power law:

$$\sigma \propto \dot{\varepsilon}^{\,0.23}$$

for parallel-plate compression, compaction over a filter, and for extrusion through a cylindrical die. Since there is no flow of liquid relative to the solid during extrusion, inhomogeneity in the flow cannot account for this power law behavior at low strain rates ($10^{-1}s^{-1}$).

List of Patents and Publications Resulting From this Work

Patents

1. M.C. Flemings and F.E. Goodwin, "Method for Making Metal Alloy Compositions and Composition," U.S. Patent No. 4,295,896, October 20, 1981.

2. M.C. Flemings and A.L. Lux, "Process for Purifying Metal Compositions," U.S. Patent Application No. S.N. 247-500.

Publications

1. A.L. Lux and M.C. Flemings, "Refining by Fractional Solidification," Metallurgical Transactions B, Vol. 10B, March 1979, pp. 71-78.

2. A.L. Lux and M.C. Flemings, "Refining by Fractional Melting," Metallurgical Transactions B, Vol. 10B, March 1979, pp. 79-84.

3. F.E. Goodwin, P. Davami and M.C. Flemings, "Strengthening of Wrought Aluminum Alloys by Fractional Melting," Metallurgical Transactions A, Vol. 11A, November 1980, pp. 1777-1787.

4. M. Suery and M.C. Flemings, "Effect of Strain Rate on Deformation Behavior of Semi-Solid Dendritic Alloys," accepted for publication.

5. D.A. Pinsky, P.O. Charreyron and M.C. Flemings, "Deformation of Semi-Solid Dendritic Alloys at Low Strain Rates," submitted for publication.

List of Participating Personnel with Advanced Degrees

Students	Degree
F. Goodwin	Sc.D., February 1979
M. Gungor	S.M., February 1979
D. Pinsky	S.M., to be granted, 1981.
A. Lux	Sc.D., February 1978
D. Smith	S.B./S.M., June 1979
A. Mortensen	

Staff

M. Flemings
M. Suery
P. Davami
P. Charreyron

Appendix

List of patents and papers from M.I.T. on Rheocasting emanating from the original ARO sponsored work or from subsequent, more applied research programs sponsored by other government agencies.

Patents

1. M.C. Flemings, R. Mehrabian, and D.R. Geiger, "Methods of Refining Metal Alloys," Patent No. 3,840,364, issued October 8, 1974.

2. M.C. Flemings, R. Mehrabian, and R.G. Riek, "Continuous Process for Forming an Alloy Containing Nondendritic Primary Solids," Patent No. 3,902,544, issued September 2, 1975.

3. M.C. Flemings and R. Mehrabian, "Metal Composition and Methods for Preparing Liquid-Solid Alloy Metal Composition and for Casting the Metal Compositions," Patent No. 3,936,298, issued February 3, 1975.

4. M.C. Flemings, R. Mehrabian, and D.B. Spencer, "Composition and Methods for Preparing Liquid-Solid Alloys for Casting and Casting Methods Employing the Liquid-Solid Alloys," Patent No. 3,948,650, issued April 6, 1976.

5. M.C. Flemings and R. Mehrabian, "Metal Composition and Method for Preparing Liquid-Solid Alloy Metal Composition and for Casting the Metal Compositions," Patent No. 3,951,651, issued April 20, 1976.

6. M.C. Flemings, R. Mehrabian, and D.B. Spencer, "Liquid-Solid Alloy Composition," Patent No. 3,954,455, issued May 4, 1976.

7. M.C. Flemings and K.P. Young, "Method for Determining the Suitability of Metal Compositions for Casting," Patent No. 4,011,901, issued March 15, 1977.

8. M.C. Flemings, K.P. Young, and R.G. Riek, "Method and Apparatus for Forming Ferrous Liquid-Solid Metal Compositions," Patent No. 4,089,680, issued May 16, 1978.

9. M.C. Flemings, R.G. Ried, and K.P. Young, "Method for Forming High Fraction Solid Metal Compositions and Composition Therefor," Patent No. 4,108,643, issued August 22, 1978.

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2. R. Mehrabian and M.C. Flemings, "Die Casting of Partially Solidified Alloys," Trans. A.F.S., Vol. 80 (1972), pp. 173-182. (Also Die Casting Engineer, July-August 1973, pp. 49-59.)

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4. E.F. Fascetta, R.G. Riek, R. Mehrabian, and M.C. Flemings, "Die Casting Partially Solidified High Copper Content Alloys," Die Casting Engineer, September-October 1973, pp.44-45. (Also, A.F.S. Cast Metals Research Journal, Vol. 9, No. 4 (1973), pp.167-171; Trans. A.F.S., Vol. 81 (1973), pp. 95-100.)

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