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Author

Gilman, J.J.

Publication Date

1986-06-01

Lawrence Berkeley Laboratory

UNIVERSITY OF CALIFORNIA

Materials & Chemical Sciences Division

Center for Advanced Materials

Invited talk presented at the Royal Society
Discussion Meeting on Technology in the 1990's:
The Promise of Advanced Materials, London,
England, June 4-5, 1986, and to be published
in Philosophical Transactions of the
Royal Society, Series A

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J.J. Gilman

June 1986

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RAPIDLY SOLIDIFIED MATERIALS

John J. Gilman
Center for Advanced Materials
Lawrence Berkeley Laboratory
University of California
Berkeley, California 94720

ABSTRACT

Although rapid solidification has been used for generations to refine the grain-sizes of materials, its effects are most significant when it succeeds in converting the liquid-state into a glass. This is because homogeneous materials can be made in this way that have distinctly different compositions, as well as different molecular structures, than traditional materials. The liquids of interest are alloys. They may consist of two or more metals, two or more polymers, two or more ceramic compounds, or other combinations. When they have been converted into glasses they may be useful in that form, or the glass may be the precursor to devitrified forms that cannot be created in any other way. Such materials often have unusual combinations of chemical and physical properties including strength, magnetic hardness or softness, electric response, catalytic ability, and corrosion resistance.

RAPIDLY SOLIDIFIED MATERIALS

Metallurgists have known for a long time that metals become harder when they are quenched from the melt rather than slowly cooled. Usually this has been associated with grain-size refinement, but is now known to involve metastable solute concentrations as well. Similarly, the properties of high polymers can be changed markedly by quenching them fast enough to suppress crystallization. This sometimes makes them transparent, and often stiffer. The effects are most dramatic for liquids that would ordinarily separate into two or more phases if they were more slowly cooled. Through fast cooling they are suspended in metastable states.

The metastability that follows fast cooling is associated with high concentrations of structural defects such as grain-boundaries, or of non-stable chemical groupings that are held in suspension by the sluggishness of the kinetic processes in the material.

For the materials engineer it is the physical and chemical properties of these metastable structures that are of primary interest. The fact that they may have been produced by means of rapid solidification is secondary. On the other hand, for the businessman, the production process is of key interest because it may determine the relative manufacturing costs; and the faster the process the lower the cost, other factors being equal.

Observations of the effects of rapid versus slow solidification were made in antiquity. However, the range of rates has been extended in recent times, particularly at the high end. Rates less than about $10\exp(-5)$ deg/sec are impractical for experimental work. Until about 1960, rates above $10\exp(+5)$ deg/sec were rarely observed. Then, following the early work on splat-cooling (Klement 1960), there were concerted efforts to increase the maximum rates. Currently,

by heating surfaces with picosecond pulses of light followed by self-quenching, rates as high as $10 \exp(+12)$ deg/sec have been achieved (Lin 1984).

At very low cooling-rates liquids tend to transform to solids across a single interface. Individual crystals result. At higher rates while one crystal is growing others become nucleated. Polycrystals result. The higher the rate, the more nucleation occurs and the smaller the grain-size of the resulting polycrystal. At very high rates, there is insufficient time for crystals to nucleate and grow so the structure of the liquid is retained, and the substance is called a glass.

Glasses are often said to be "amorphous" which in Greek means "without structure", but this is misleading because they usually contain short-range-order. They inherit this from their mother liquids which usually contain associations of atoms or molecules. Even one-component liquids are often polymerized.

As solidification rates have increased, grain-sizes have decreased and metastable solute concentrations have increased. These trends have been evolutionary. For example, chill blocks have been used in the manufacture of railway wheels from cast-iron for over a century.

However, when alloys were quenched by Klement, Willens, and Duwez at rates that completely suppressed crystallization in them, a revolution began. At first this involved scientific studies of metastable alloy phases, including glasses. The specimens that were produced had little or no technological interest because they were misshapen, often brittle, and often composed of costly raw materials. Then, in 1972, H. Chen and D. Polk (1973) spun the first ductile ferrous glasses at the laboratory that I organized for the Allied Chemical Corporation (now the Allied Signal Corporation).

A photograph of Chen's first specimen is shown in Figure 1. Its composition in atomic per cent is 39Fe, 38Ni, 14P, 6B, 3Al; and it weighed about five grams. As of this writing, the specimen remains ductile. At present, thousands of metallic glasses have been made. In production lines, ribbons are made at lineal speeds up to 6000 ft./min. and in continuous lengths many miles long from ton-sized heats of metal.

It should be noticed that this revolution has been based on the confluence of several elements, not just one. In order to introduce a new material to the world of metallurgy it was necessary to invent not just any glass, but a glass with a relatively inexpensive composition combined with an inexpensive method for producing it. Furthermore, it was critical that it have ductility, and that there be a multitude of potential applications for it.

The revolution has been markedly strengthened by the fact that in the early stages of devitrification, glasses transform into microstructures that cannot be realized in any other way (or at least not easily realized). This has led to remarkable structural materials (Ray 1983) and to exceptional iron-neodymium-boron permanent magnets (Croat 1984). Thus glasses are not only of interest for themselves, but also as precursors for special kinds of polycrystals.

Some people think that this subject has been exhausted by the hundreds, if not thousands, of projects that have been undertaken by people interested in it. I think this is a pessimistic position. The subject has so many variations and ramifications that I think continued work will lead to many new scientific insights and technological opportunities.

While preparing for this lecture, I reviewed some of the many writings about the rapid solidification of metals, and tried to find information about this process in the fields of ceramics and polymers. I could find very little in these latter fields although I did learn that a major abrasive is rapidly cooled from its liquid in order to refine its grain size. One of its manufacturers is the Norton Company and it has a mixed aluminum, zirconium oxide composition. Its fine grain-size enhances its toughness. Laboratory studies of ceramic materials have been occurring,

however (Revcolevschi 1981).

Applications of quenched polymeric materials have escaped me, although laboratory research by H. S. Chen and others has been done.

As I already stated I think that the development of fine-grained, non-equilibrium, metallurgical materials through rapid solidification has been an evolutionary process. In recent times it has taken the form of improving the compositions and grain-structures of the powders that are consolidated to make tools, jet-engine disks, and other structural parts. This has made very substantial improvements in performance, but it has not added a new revolutionary branch to the field of metallurgy as has the technology of metallic glasses. Therefore I shall concentrate on the latter in the remainder of this lecture.

Basis of the Novelty of Metallic Glasses

The novelty of metallic glass technology is based primarily on three factors: composition; molecular structure; and fast processing. These factors are not independent of one another, but they can be discussed separately.

Consider composition first. The compositions that form glasses readily had rarely been used in the past because relatively slow cooling of them usually led to brittle substances. One class of examples that illustrates this point is the transition metal-boron alloys. In these alloys relatively small concentrations of boron leads to brittleness if the alloy is cooled slowly. But if the alloy is quenched to the glassy state ductility exists at atomic concentrations of boron that are 10 to 100 times as great as in boron-containing steels. Thus, glassy alloys can have dramatically different compositions from the crystalline alloys of the past.

Next, consider molecular structures. As I mentioned earlier, the liquids from which glasses are formed contain well-defined short-range-order. This has been demonstrated by examining the distribution of eutectic (glass-forming) compositions in a large number of binary systems. It is found that eutectics are clustered at specific integral atomic-ratios (Gilman 1978). Thus the short-range-order in a glass is characteristic of the mother liquid.

The characteristic structure of a liquid is different in most cases from the short-range structure that would be found in other antecedents of "amorphous" alloys, such as: gases prior to condensation; plasmas during deposition; electrochemical solutions prior to plating; and so forth. Thus the short-range molecular structures that metallic glasses inherit from their mother liquids are unique. They may well result in unique physical properties, although this does not necessarily follow.

Finally, consider fast-processing. A ribbon that is 3 mils (0.074 mm.) thick and 4 in. (98 mm.) wide being cast at 3000 ft./min. (9882 m./min.) corresponds to a casting rate of 3.6 tons/hr. (3300 kg./hr.). When one considers that the product is in its final form (and has gone from liquid to metal to it in one step), plus the fact that lightweight equipment is used (because the resistance to the shape change is only the low viscosity of the liquid metal), this is indeed a unique, revolutionary manufacturing process.

In this process, the liquid alloy is first melted in a large crucible and then poured into a tundish which delivers it to a specially shaped nozzle (Figure 2). The nozzle lays it onto a rapidly-rotating water-cooled wheel which it partially wets. As it travels with the wheel it loses heat quickly and solidifies into the glassy state. Then the solidified ribbon leaves the wheel, it passes through guides and monitoring sensors, and then onto a windup sheave. If the operating mode is quasi-continuous, an automatic mechanism changes sheaves as they become full, and a sequence of melting crucibles replenishes the tundish. Signals from the monitoring sensors provide

the means for feedback control of the process.

This fast-casting technology (developed at Allied Signal) for producing metallic glass ribbons can be applied to other materials, of course. For example, it has already been applied to polycrystalline brazing tapes.

Development of a satisfactory quasi-continuous process required many inventions of new engineering elements. These include: planar-flow casting nozzles; cooling systems for intensely heated quenching-wheels; mechanisms for catching ribbons moving at high velocities, threading them into rotating sheaves and "doffing" the sheaves as they become full; sensors and systems for feedback control; and methods for handling and metering liquid alloys.

The importance of fast processing can be appreciated by considering a demand chart for metals. The demand (#/yr) for metals is strongly correlated with their unit prices (\$/#) as indicated in Figure 3. The annual revenues that are generated by the sales of any given metal are strongly dependent on the unit price as indicated by the dashed lines in the figure.

This may be clarified by replotting the data to show the dependence of revenues on prices as in Figure 4. The powerful effect of price on dollar volume that this figure indicates provides a forceful motivation for seeking low manufacturing costs.

It is not a secret, of course, that fast processing tends to minimize manufacturing cost. But the magnitude of the effect in this particular case does not appear to be widely appreciated so it may be useful to consider some numbers. Suppose that a quench-casting unit produces one-inch ribbon at a rate of one ton/hr. and the unit costs \$100,000 at a time when the interest rate is 8 per cent. Further suppose that the unit is leased (in order to minimize the arithmetic). Then the direct rental charge for a one-shift operation and a 40 per cent duty-cycle is about 0.01 \$/#. Compare this with a hot-pressing unit that costs the same amount and produces about 10# of product per shift. In this latter case, the rental charge will be about 10 \$/#, or about 1000 times greater.

In the first case, the capital rent per unit of product is a small fraction of the raw materials cost. In the second, it is a multiplier of the raw materials cost. It seems clear then which process is more desirable.

The other primary manufacturing costs are: energy, labor, and equipment expendables such as nozzles. They act to reinforce the conclusion above. The efficient use of capital equipment in most processes is a dominant factor in determining costs, and quench-casting uses equipment very efficiently.

Translation of Novel Properties into Applications

New materials find their way into applications either by substituting for older materials in established applications, or by allowing devices to be invented that exploit their new properties. Then the new devices substitute for older ones. In either type of substitution, the new must have more utility than the old (that is, be more cost-effective). Otherwise there is no driving force for the substitution and it will not occur.

Fast processing combined with some unusual combinations of properties is what has made metallic glasses very attractive for a variety of applications. For example, they combine extreme hardness with high corrosion resistance. This makes them attractive for cutting devices such as razor blades and scalpels. Also, they combine magnetic softness with mechanical hardness. This is an excellent combination for tape-recording heads. Some alloys combine high magnetostriction

with high strength so they make effective transducers. And so on. So many actual and potential applications have been reported that it is difficult to summarize them succinctly, but an attempt will be made here, starting with magnetic devices.

Ferromagnetic glasses are very easily magnetized because magnetic domain-walls move through them with extraordinary ease as shown by the listing of mobilities for some typical materials in Figure 5. The mobility for the glass is 500 times greater than for the standard Fe-Si material. This is why these glasses are such an extraordinary advance in the field of soft magnetic materials.

a. Transformers

When the mobility described above is combined with the other properties of magnetic glasses and made into an electric power transformer, the core losses are markedly reduced. Data for a variety of prototypes made by five different manufacturers and in five different sizes are shown in Figure 6. For the small sizes, the difference between the standard material and glass is a factor of four, while at large sizes (say 100 kVA) it becomes a factor of ten, corresponding to the intrinsic characteristics of the materials (Figure 7).

Since there are nearly 40,000,000 power distribution transformers in the USA, the total losses are about 35 billion KWH annually. Glass cores could save 23 billion KWH of this. At an average cost of \$0.05/KWH this is 1.2 billion dollars per year. In other countries where the cost of electric energy is higher, the savings would be comparable or greater. Naturally, this has generated intense interest on the part of the electric utility industry. Numerous test transformers have already been placed at field locations and are being observed. For a set of 25 distribution transformers, no significant aging effects were observed over a two year period (Bailey 1986).

In the second generation of field testing, the Electric Power Research Institute commissioned the General Electric Company to manufacture 1000 distribution transformers (25 KVA size) made with glass manufactured by the Allied Signal Company. These are being distributed to the utility companies that support EPRI for two year field tests (Smith 1985).

The largest transformer made to date is a 500 KVA unit made by the Westinghouse Corporation. Its reported core losses are 200 W, as compared with 1000 W for a similar unit with a conventional steel core.

The magnetic cores of electric motors also convert large amounts of their input energy into heat. It has been shown that this could be substantially reduced if metallic glass cores were used in them (Raskin 1983). However, this will not happen unless some difficult fabrication problems are overcome.

For power frequencies up to about 100 KHz metallic glasses make superior small transformers. This has led to their use in switching-mode power-supplies (Coutant Electronics, Toshiba), tape-recording heads (Sony, Kenwood), aircraft transformers (Allied Signal), pulse suppression chokes (Toshiba), current-metering transformers, ground-fault interrupters, transformer turns-ratio tester (Biddle Instruments), high-speed welder (Mitsubishi), and a variety of other small transformers (Raskin 1983).

If very high magnetic fields are applied to selected metallic glasses their magnetizations can be reversed in as little as 100 nano-sec. Thus, they are useful as the cores of pulse transformers and magnetic switches. These can be very large (up to several tons in weight) and are used in ion accelerators, ultra-violet lasers (excimers), and radar modulators. They can be used to switch pulses in the multi-terawatt range of power; that is, to switch one million amperes at two million volts (Smith 1985). A new particle accelerator called Hermes IV that will be built at the Sandia

National Laboratory is scheduled to have 110 saturable reactor cores made of metallic glass. Since each will weigh about 300 kg. this machine will use about 33,000 kg. of metallic glass.

The magnetization loop for an easily magnetized metallic glass is quite "square". That is, under the influence of a small applied field the magnetization rate rises rapidly to nearly its saturation level and then remains nearly constant. In other words the magnetic response of the material is highly non-linear. A result is that if the material is exposed to electro-magnetic radiation of a particular frequency, it reradiates at several harmonics in addition to the fundamental frequency. This makes it useful for surveillance devices because the distribution of harmonics that is reradiated provides a "signature" that is relatively easy to detect.

An additional feature can be used to advantage in surveillance devices. This is the mechanical hardness of the material which has very low acoustic-attenuation as a corollary. In other words, the glass "rings" for a long time after it is set into mechanical vibration. Thus excellent mechanical oscillators can be made from it, and this allows its response to incident electro-magnetic radiation to be sharply tuned. Through tuning, its reradiation signature can be designed, and the sensitivity of a device markedly increased (Anderson 1985).

b. Magnetic Shielding

The high magnetic permeability of metallic glass is the basis of this application. Cobalt-bearing alloys of low magnetostriction are particularly effective. Shields may be constructed by weaving ribbons into a fabric, or by wrapping cables with ribbon in a helical configuration, or by other methods. Shielding ratios (field outside/field inside) of 100 are achieved with double helical windings on a cable (60 Htz). For the same amount of shielding this is about 8 times better than previous materials (Hilzinger 1985).

c. Transducers/Sensors

Various combinations of the electrical, magnetic, and mechanical properties of metallic glasses have been used as the bases of transducers. There is insufficient space here for a review of them, but a listing (no doubt incomplete) will be made in order to indicate the large scope of applications (Mohri, 1984, 1985; Makino 1985; Matsuki 1985).

1. High-output tape-heads (both audio and video)
2. Photo-cartridges
3. Fast-response magnetometers
4. Delay lines
5. Displacement, force, or torque transducers
6. Strain gauges
7. Rotational speed sensors
8. Fuel-injector controls
9. Temperature sensors
10. Vibration sensors
11. Electro-magnetic radiation meters
12. Height gauges

- 13. Microphones
- 14. Data-tablets
- 15. Cloth inductor

d. Joint Formation (brazing)

One of the earliest applications of metallic glasses was the manufacture of brazing tapes for joining parts of jet-engine components. The basis of the application is that the new glassy forms of the alloys that are used for this purpose are ductile, unlike the previous crystalline forms which are very brittle. Ductile tapes are much easier to manipulate, and they make superior joints, compared with the powders that were used previously.

The scope of this application area has been expanded by the development of ductile polycrystalline tapes of a variety of compositions, and it will probably continue to expand as the quench-casting technology is developed to include still more alloys. At present at least twenty-five compositions are offered commercially. They are grouped into families. One of these is nickel-based tapes for brazing high-temperature alloys to make engine components, heat exchangers, and food processing equipment. Another is nickel-palladium tapes to replace gold-based ones in aerospace and power-electronics brazes. A third is copper-based alloys to replace "silver-solders" for the joining of copper- and iron-based alloys. The glassy tapes use lower cost components and they melt and flow more uniformly than the materials they replace (DeChristofare 1985).

Even ordinary solder benefits from the quench-casting process. The joints made using quenched solders have finer-grains, better homogeneity, and smaller shrinkage-voids than ones made with conventional solder.

e. Mechanical Components

Some of the mechanical properties of metallic glasses are outstanding. For example, they have the highest biaxial tensile strengths of any known material. So they should make useful composite structures (Gilman 1984). Also, as a result of their homogeneity, laboratory tests have demonstrated that they make superior cutting edges. And very strong wires can be made from them. Industrial applications based on these properties have been slow in developing. The principal reason may be fabrication difficulties. For example, such things as the hardness of the glass which rapidly wears stamping dies, and finding compatible adhesives for making composites that can withstand cyclic loadings, and so on. It is very encouraging for the future of these applications that excellent fatigue resistance of the glasses themselves have been reported recently (Hagiwara 1985).

f. Chemical Components

One application of metallic glasses in chemical processing is for filters in high-field, high-gradient filtering systems. These are used to remove paramagnetic iron oxides from clays and waters which are otherwise discolored by them.

Other applications such as using glasses as catalysts, and as corrosion resistant coatings, have encountered fabrication difficulties much as the mechanical applications have. However, it

appears that these are gradually being overcome (Cocke 1986).

Descendants of Glasses

Two new branches of the metallurgist's art have descended from the early work on glasses. One is the fast continuous direct-casting of ribbons and wires. The other is the use of glasses as precursors for fine-grained polycrystalline metals. I will not consider the first of these further since I have already presented some of the arguments in its favor.

In general the microstructural state that is reached by devitrifying a glass is not the same as the one that is reached by rapid solidification to a fine-grained polycrystal. In the latter case crystallization occurs in the presence of a large thermal gradient, whereas in the former case it may occur under nearly isothermal conditions. In various practical situations the results may be virtually indistinguishable, but not always.

The first work that I know of in which exciting new materials were derived from glasses is that of Ranjan Ray at Allied Signal. He quenched ferrous alloys containing about half as much boron as usual into the glassy-state. The resulting ribbons were then warmed to embrittle them and while in the brittle state they were fragmented to form a coarse powder. The powder was consolidated into cylinders by hot-pressing, or hot-extrusion. These devitrified materials were strong, tough, and heat-resistant. They were, and are, quite remarkable (Ray 1983). Their microstructures consist of tiny grains about one-tenth micron in size, stabilized by very small borides about one one-hundredth micron in size at the grain boundaries. This led to the development of Ni-Mo-Cr-B materials with attractive properties for die-casting molds and hot-working dies (Chang 1985).

Even more exciting was the discovery by Croat and Herbst at the General Motors Research Laboratories of a new class of permanent magnet materials, based on Fe-Nd-B compositions (Croat 1984). They initially discovered that quenching a 60Fe-40Nd alloy at an optimum intermediate rate yielded material with a coercive force of 7.5 kOe. Later it was found that a full quench to the glassy-state followed by tempering to initiate devitrification gave essentially the same result. Also, it was later found that boron additives improved the hard magnetism, and that alignment of the devitrification products by hot-working further improved the properties.

Parallel work at Sumitomo Special Metals Company done by sintering appropriate powders produced similar high performance material. However, it appears that the quench-casting method is more cost-effective.

A figure-of-merit for hard magnets is energy product. Values as high as 50 MGOe have now been achieved for Fe-Nd-B magnets. This may be compared with 10-20 for the best previous material, samarium-cobalt.

This material is already in production for use in the small electric motors used for actuators in automobiles.

A series of descendants has followed these early ones. In all of them, beneficiation occurs as a result of grain refinement and fine-scale homogeneity, and increased solute concentrations. From these, higher strengths, toughnesses, increased thermal stability, and enhanced corrosion

resistance follows. These descendants include:

1. Aluminum-iron alloys for use at high temperatures (Skinner 1985)
2. Magnesium-RE corrosion resistant alloys (Das 1986)
3. Tungsten and uranium alloys for ballistics (Davis 1986)
4. Titanium alloys (Kubel 1986)
5. Shape-memory alloys (Li 1985, Eucken 1985)
6. Precipitation-hardened stainless steels (Ray 1986)
7. Iron-silicon transformer steels (Warlimont 1985)
8. Nickel aluminide with boron (Taub 1985)

Forecast

The future of this field is likely to be just as exciting as the recent past. Much of the territory remains unexplored although some strong themes were established early in the development period.

First of all, relatively little is known about the structures of glass-forming liquids prior to the time of quenching. It is to be expected that their structures will vary with temperature above the liquids; sometimes sluggishly. Also, it is known empirically that relatively small additions of specific elements can change the liquid behavior observably.

Through adjustment of the liquid composition relative to the phase equilibria heterogeneous structures can be generated. An example, first shown by D. Narasimhan, was obtained by cooling a nearly eutectic liquid below its liquidus which caused small particles of precipitate to form. This mixture was then quenched to convert the remaining liquid into glass. The result was hard particles embedded in a ductile glass. This technique has not been fully exploited.

Composition itself has a primary influence on properties. So many compositions have been tested for their glass-forming propensities and subsequent properties that it seems at first sight that there is little room in which to explore further. This may be true for most of the potentially interesting binary systems, but it is certainly not true of ternary, quaternary, and higher order systems. Therefore, searches for important new compositions should continue to be rewarding.

There are various opportunities for the development of new, or improved, processes. Quench-casting itself can be further improved, and better methods for consolidating ribbons are needed. Coating techniques, shell-casting, and other processes can be improved.

Perhaps the most promising area of all for the future, is that of using glasses as precursors for fine-grained polycrystalline materials. These may have unusual compositions, microstructures, and properties. They already form the bases of permanent magnet materials, as well as strong materials that are stable at high temperatures. However, only a relatively few compositions have been explored. Also, much more can be done to determine the effects of applied fields other than temperature. For example, stresses and magnetic fields. Gradients may also induce new microstructures. Thus directional devitrification will put a strong texture into the product and this may enhance desired properties in some cases.

In the 13-14 years that have passed since the first ductile ferrous-glass was invented, the annual production rate has increased from about 2 #/yr. to 2-20 x 100,000 #/yr. This corresponds to a growth-rate of 85-100 per cent per year. Since there is no reason to expect that

this growth-rate will drop in the near future, it is easy to see that this technology indeed represents an important new branch of metallurgy.

Acknowledgments

Several people were very helpful to the author during the preparation of this paper. They provided their opinions of the status of the field and copies of a large number of preprints and reprints. Listed alphabetically, they are: L. A. Davis, M. C. Flemings, R. I. Jaffe, R. W. Lee, Y. Makino, I. Ogasawara, T. Sato, C. H. Smith, D. R. Uhlmann, and H. Warlimont.

Preparation of this manuscript was supported in part by the U. S. Department of Energy under contract No. DE-AC03-76SF00098.

Figure Captions

- Figure 1 Piece of the first ductile, ferrous glass made by H. S. Chen. The composition is $\text{Fe}_{39}\text{Ni}_{38}\text{P}_{14}\text{B}_6\text{Al}_3$.
- Figure 2 Quasi-continuous quench-casting manufacturing line. Note wrapping of ribbon on casting wheel for traction. Flow is controlled through combination of liquid head and gas pressure in tundish (details not shown).
- Figure 3 Demand chart for structural metals.
- Figure 4 Dependence of revenues on price for structural metals (data from Figure 3).
- Figure 5 Comparison of magnetic domain wall mobilities in various materials.
- Figure 6 Energy losses in transformers of various sizes made by various companies. The decreases achieved using metallic glass cores is dramatic.
- Figure 7 Comparison of intrinsic core losses between standard magnetic steel and metallic glass.

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FIGURE 1

FIGURE 2

XBL 865-2007

FIGURE 3

XBL 865-11129

Structural Metals – 1980

XBL 865-2005

FIGURE 4

Material	Domain-wall Mobility $\left(10^{-3} \frac{\text{cm}^3}{\text{d-sec.}} \right)$
Fe-Si Crystal	4
Ni-Fe Wire	6
Fe Crystal	40
Ni-Fe Glass	2000

XBL 865-11128

FIGURE 5

XBL 865-10056

FIGURE 6

XBL 865-10095

FIGURE 7

This report was done with support from the Department of Energy. Any conclusions or opinions expressed in this report represent solely those of the author(s) and not necessarily those of The Regents of the University of California, the Lawrence Berkeley Laboratory or the Department of Energy.

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UNIVERSITY OF CALIFORNIA
BERKELEY, CALIFORNIA 94720*