

SOME IDEAS ON THE CHOICE OF DESIGNS AND MATERIALS FOR COOLED MIRRORS*

M.R. Howells

Advanced Light Source
Accelerator and Fusion Research Division
Lawrence Berkeley Laboratory
University of California
Berkeley, CA 94720, USA

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Advanced Light Source, Lawrence Berkeley Laboratory, 1 Cyclotron Road, Berkeley, CA 94720,
USA.

1.0. Introduction

It is a pleasure to join in this celebration of the achievements of Klaus Halbach and to record my personal gratitude to him for the many insights I have gained both via his written and spoken presentations and through personal contacts. I am happy to contribute a section to this book which I believe will be a very unusual document. It offers some special opportunities to write in a more subjective and speculative way than do the conventional avenues of scientific publication and I plan to take advantage of that to express some of my views on how the fabrication of future synchrotron beam-line optics ought to be approached.

Many of the most interesting new ideas for beam-line mirrors, especially those with a promise of low costs, involve metals. Historically these materials had posed certain problems which have been overcome in recent times¹ to the extent that the initial complement of Advanced-Light-Source (ALS) beam-line optics were made of metal and have met their specifications. To go further along that road we need to get more interested in the metallurgical issues involved in making high-quality metal mirrors. I will recount the results of some of my investigations into these materials questions and will try to draw on some of the experiences and achievements of other communities which have hitherto had only limited contact with synchrotron radiation researchers.

Most of the cooled mirrors used in synchrotron beam lines have fairly simple designs based on cooling channels of *uniform* cross section. These are sufficient for many purposes as we shall see but for the most challenging heat loads a more sophisticated type of design, the cellular-pin-post system² employing a very complex coolant flow path, has been developed. We will return to the cellular-pin-post system later but first we consider the theoretical description and analysis of a simpler mirror with uniform cooling channels.

2.0. Theoretical analysis

The basic layout for cooling a heated surface is a layer of identical side-by-side cooling channels below the surface as shown in Figs. 1 and 2. There is a thin, flexible layer of material above the water channels (the "hot wall") which has a temperature gradient across it. Underneath the water channels is a large thick block; the main mirror substrate, which has a much greater stiffness (thickness) than the hot wall. The principle of the design is to remove all the incoming heat via the water so that the whole lower substrate should be at a uniform temperature close to that of the water. With this type of design the gross bending can be reduced as much as desired by increasing the ratio of the thickness of the main substrate to that of the hot wall. The main distortion is therefore a swelling of the mirror perpendicular to its surface which "maps" the shape of the incoming power distribution. This distortion has been calculated using simple heat-transfer theory by the present author and was reported in detail in an earlier paper³. Only the general trends and insights provided by that calculation will be presented here. Later we will try to use the understanding provided by the theoretical analysis as a guide in developing mirror designs and in choosing substrate materials.

Following the arrival of an absorbed power density $\dot{Q}''(y,z)$, heat will flow from the top surface of the mirror to the water with a conductive temperature drop across the hot wall and a convective temperature drop across the solid-water interface. These are determined by a conductivity, k , and a convective heat transfer coefficient, h (defined as the heat transfer per unit area per unit temperature drop). For a mirror made of a good conductor, the temperature drop across the coolant interface usually dominates and we speak of convection-limited heat flow. Conversely, for poor conductors, we expect conduction-limited heat flow.

To make a quantitative analysis we assume the coolant geometry shown in Fig. 2 which is easy to manufacture, close to optimum in performance and amenable to calculation. The hot-wall thickness should be enough to provide mechanical strength to resist the forces due to polishing and water pressure. We consider the rectangular segments of mirror material between the water channels to be "cooling fins" for the hot wall. The properties of such structures have long been analyzed in the engineering literature^{4,5}. For suitably good conductors, a fin can remove much more power per unit area per unit temperature difference (relative to the water) than can a direct solid-water interface.

We assume that the hot wall is restrained in two dimensions, the length and width directions of the mirror, and is unrestrained in the thickness direction while the fins are restrained in only one dimension, their length. This can be accounted for by defining two new expansion coefficients $\alpha_1 = \alpha(1 + \nu)$ for the fins and $\alpha_2 = \alpha(1 + \nu)/(1 - \nu)$ for the hot wall, where ν is Poisson's ratio.

For a given water-flow rate one can calculate h and thence, via the standard analysis of fins⁵, obtain the temperature distribution³

$$T_0 = \frac{\dot{Q}''}{h \left[\frac{2FH\eta_f}{w} + 1 - F \right]} \quad (1)$$

where we have used a fin efficiency factor $\eta_f = \tanh(mH)/mH$ with $m = \sqrt{2h/kw}$ and the rest of the notation is given in Figs. 1 and 2. The surface-height error is then

$$x(y,z) = \dot{Q}''(y,z) \left\{ \frac{\frac{\alpha_1}{h} H\eta_f + \frac{\alpha_2}{h} t}{\frac{2FH\eta_f}{w} + 1 - F} + \frac{\alpha_2}{k} \frac{t^2}{2} \right\}. \quad (2)$$

Equation (2) represents our analysis of the "mapping" distortion of the mirror. We may summarize it as $x = A\dot{Q}''$ or $dx/dy = Ad(\dot{Q}'')/dy$ in which case the quantity A , the height error per unit power density is a measure of the goodness of the cooling and is known in some communities as the "worm factor". The first term in the brackets describes the growth in the length of the fins. The second term describes the growth in thickness of the hot wall due to the rise in the temperature of the fin-hot-wall interface resulting from the convective heat transfer. The third term describes the growth in thickness of the hot wall due to its conductive top-to-bottom temperature difference (ΔT_{0M}). The two h terms will dominate for convection-limited heat flow and the k term for conduction limited heat flow.

The significance of the so-called "distortion figure of merit" k/α becomes clearer. It determines (via the third term of equation (2)) the growth of the hot wall thickness due to its conductive temperature drop which is the main effect in conduction-limited heat flow. On the other hand the importance of α/h in determining the size of the first two terms which dominate in convection-limited heat flow is also evident. Thus k/α is not a true, simple figure of merit in this situation. In a mirror made of glidcop, for example, the last term, which contains k/α , is likely to account for only a few per cent of the total distortion.

In Ref. 3 the above theory was applied to an example glidcop mirror design with a 2-mm hot wall and both channels and fins having a $1 \times 6 \text{ mm}^2$ cross section. The x-ray source was a ESRF wiggler which deposited a power density of 0.46 W/mm^2 at the mirror. The following conclusions could be made:

- Even in such a high powered beam, the slope errors were in the arc-second or sub-arc-second range for all the materials considered so that this type of design is useful.
- The slope errors were best for invar, silicon carbide and silicon. They were good for molybdenum and beryllium and only fair for glidcop, aluminum and stainless steel. Fused silica had to be ruled out because of excessively high surface temperatures.
- Both the temperature and height values obtained from equations (3) and (4) agreed with those from finite-element calculations using the RASNA code within less than 5 per cent.
- For stainless steel, invar and fused silica the heat flow was conduction-limited, for the other materials it was convection-limited.
- If the materials are placed in slope-error order of merit, they are with few exceptions in *inverse order* of ease of fabrication and general convenience.

We do not wish to concentrate on the details of these calculations which are covered fully in Ref. 3. Rather we wish to explore the consequences of the insights that the theory provides for mirror design and for materials choices.

3.0. Cooling channel design considerations

The optimum design of the fin structure depends on whether the heat flow is conduction- or convection-limited as indicated by the values of the thermal resistances $\theta_h = T_0 / \dot{Q}''$ and $\theta_k = t / k$. For conduction-limited cases it is less important to have good fin design, in fact the fin advantage tends to disappear in such cases, but it is very important to have a thin hot wall both for better slope errors and lower temperatures. To the extent that the lower conductivity materials are useful, this is the key to using them. To have a thin hot wall it is also necessary to keep the channel width small enough compared to the hot wall thickness to avoid "print through" of the underlying structures but for materials like steel it should be quite practical to use 0.5-1 mm walls with appropriate care. The thin hot wall is especially significant for invar because the reduction of the mirror surface temperature prevents a growth in the expansion coefficient in addition to the other effects. On the other hand for convection-limited cases the hot-wall thickness is less important than good fin design. This essentially means increasing the effective area of the solid/water interface which implies enlarging the *number* of fins which again means narrow fins and channels. A limit to this is set by the desire to maintain turbulent flow at the available water pressure. Some examples of parameters for reasonable design scenarios are given in Ref. 3.

We need to consider where the ultimate limits lie in the most challenging cases. They break down into two major categories: (i) extreme heat loads where there is a difficulty to keep the mirror surface at a safe temperature and the coolant interface below boiling temperature (an example is the

ALS infrared beam line first mirror) and (ii) extreme surface tolerances (an example would be a projection x-ray lithography imaging mirror). For case (i) where distortion is secondary, the design will have the smallest possible grazing angle and the largest number of channels one can manufacture. The material choice will be based on high conductivity and perhaps microyield stress and glidcop would be a leading candidate. This then puts the onus on providing sufficient water pressure to achieve enough flow speed to get the required h value (h is proportional to $(\text{speed})^{0.8}$). The pressure drop needed for a given h is proportional to the flow distance through the mirror. Thus even after we have assigned the highest pressure drop we can, we still have one final recourse which is to make the flow distance through the fin system *short*. This was the strategy of Tuckerman⁶ and for many synchrotron radiation mirrors the beam footprint is narrow in one direction so a flow distance of a centimeter or two is often possible. An even better approach to this kind of extreme heat load would probably be a cellular-pin-post design² (see section 4.3) in glidcop.

For case (ii), where distortion is the pre-eminent consideration, we need to minimize α/h for convection-limited flow and α/k for conduction-limited. Thus, considering only the thermal parameters α and α/k , we find that invar (carefully processed as described later), silicon and silicon carbide become the leading candidate materials.

4.0. Choice of mirror material

4.1. General principles and overview

The question of choosing a mirror (or grating) substrate material is one of the most difficult and controversial of all the steps involved in planning a beamline. The issues have to be evaluated in the context of the prevailing requirements and tolerances but they are roughly as follows:

- Polishing: can a good optical figure and finish be obtained?
- Cooling: can a good thermal design be made?
- Engineering: can one design and fabricate the mirror to suite beam-line constraints?
- Material quality: can the material be obtained in the required quality and size and will it hold its shape over long times?
- Cost: is it within budget?

We now discuss some candidate materials in the light of these questions.

4.2. Silicon carbide

Recognition of the x-ray-beam-power problem in the 1970's lead some members of the community to the notion of silicon carbide mirrors⁷ which had originally been proposed in 1976 by Choyke⁸. In those days the prevailing material in use was glass, particularly fused silica, and it is certainly true, as we have seen, that the low thermal conductivity of glass is a fatal disadvantage for high-power applications. Once the idea of a silicon-carbide solution became known it gained momentum rapidly and has been favored in one way or another by most of the synchrotron-radiation laboratories. We will consider the trade-offs involved in using silicon carbide in some detail mainly to show that it has not provided and still does not promise a credible pathway to building the most challenging mirrors for the beam lines of the present day or the near future. What it has done, in essence, is to provide a way to extend the design style associated with glass optics from low power to medium power radiation beams. We believe that this is not an adequate return on the large integrated investment that the community has now made in silicon-carbide mirrors and that it is time to recognize that this approach to beam-line optics is being overtaken by events.

Most of the bulk silicon carbide available today is made by sintering and/or hot pressing powders of silicon carbide leading to the sintered- α and hot-pressed forms of the material which are less than 100% dense. Another type known as reaction-bonded silicon carbide is made by isostatically pressing fine mixtures of silicon carbide and graphite powders which are then siliconized in a furnace. Being 100% dense and fairly inexpensive, this material is of particular interest for ultrahigh vacuum components. It has been supplied to the synchrotron radiation community for example by British Nuclear Fuels⁹ as Refel and Carborundum Company¹⁰ as KT. The material has about a 10% excess of silicon which is non-uniformly distributed and this seems to have prevented the raw material from being used directly to make superpolished optical surfaces¹¹. The type of silicon carbide with the best optical properties (high uniformity, 100% density, small grain size) is the chemical-vapor-deposited (CVD) material which can be coated on a suitable substrate by pyrolysis of methyltrichlorosilane in an excess of hydrogen in a low-pressure CVD reactor¹². The rate of deposition is slow, generally less than 0.1mm/hour, so thick coatings are difficult and expensive. They are also prone to excessive stresses. The group at Morton International¹³ have produced some of the largest pieces of optical-grade silicon carbide and have made extensive measurements to characterize the material^{14, 15}.

For making mirrors, the preferred approach is normally to coat a fairly thin CVD layer on a substrate of one of the other forms of silicon carbide or graphite¹⁶. The advantages of silicon carbide for beam-line mirrors are:

- good values of k and α ,
- capability to be polished to a good finish,
- chemical inertness sufficient to allow acid cleaning in the event of hydrocarbon contamination,

- high specific stiffness.

Some of these advantages are lost if a graphite substrate is used. There are also a number of disadvantages to be concerned about. Optical-grade CVD silicon carbide is still a research and development material which is specially made for each application. Its physical properties are sensitive to many production parameters^{15, 17} and are not yet repeatably measured³. Only a few laboratories can make optical CVD material and the number that can make large (hard-x-ray-mirror-sized) pieces in reliable quality is even smaller. The extreme hardness of silicon carbide (about Moh 9.5) which is advantageous for resisting handling damage is a major disadvantage in working the material which becomes limited to specialists. Even more serious is the fact that the use of internal cooling of CVD-coated-substrates is so difficult that it appears never to have been attempted for a synchrotron-radiation optic. This has had the effect of locking out the silicon-carbide technology from the most challenging thermal problems which are specifically the ones for which it was introduced. Even the polishing step, although capable of good results, is several times slower and thus more expensive than for competing materials.

The closest so far to the technology needed for a silicon-carbide beam-line mirror with intensive cooling was a high-power laser mirror reported in 1982 by the group at the TRW company¹⁸⁻²⁰. This was a circular mirror with a layered structure composed of plates of pure CVD silicon carbide diffusion bonded together. The circular plates had machined cooling channels and fins similar to those in Fig. 2 and were separately fed with cooling water. The scheme appeared to be adaptable to the rectangular grazing-incidence geometry, but further study would have been needed to achieve a UHV technique for coolant connections. Altogether, the prospect for a reasonably-priced beam-line mirror by this route did not look very good and it was not pursued.

Another approach to cooling silicon-carbide mirrors which has often been used on beam lines is indirect cooling of the mirror sides via cooled pressure plates. Although such schemes can extend the usefulness of simple block-shaped mirrors toward higher heat loads, we do not believe that they are the natural way to approach the cooling problem nor that they are the way of the future. Most engineers, presented with the task of removing the heat arriving at a surface, would put coolant below the surface. This is what the designers of high-power laser mirrors have been doing for years and it is what the military do for "directed energy" mirrors. Thus we must ask ourselves, "if internal cooling is really the natural way to approach the problem, why is it that indirect (side) cooling is so often used at synchrotrons"?

In the opinion of this author it is a matter of the evolution and the commercial availability of the technical capabilities for making high-power beam-line mirrors. There is an interesting parallel with the cooling of electronic circuits. For the earliest semiconductor devices, there was no need for any dedicated cooling system and heat was removed via the connection pins. Later, cooling was applied to the board carrying the circuit and then, as the heat output increased, heat sinks and

cooling clips were clamped to the devices and cooling-fin systems were added. In recent times more serious thermal engineering has been applied and for the highest-heat-flux devices (currently radio-frequency amplifiers and laser diodes) water-cooling channels have been integrated into the device package while the Pentium computer chip has internal air-cooling channels. It would appear that the synchrotron optics technology is in the middle of a similar sequence of developments where useful incremental improvements have been achieved by switching from glass to silicon carbide or (more recently) silicon and from cooling via the mirror mountings to cooling via clamp-on structures. However, as the heat loads and distortion specifications get tougher, there is an increasing necessity to apply cooling directly to the heated object (the mirror) and close to the applied heat load. This is not necessarily more expensive than the other approaches but it leads to different designs and materials (metals and silicon will be favored) and requires that the optical, mechanical and thermal aspects of the project be integrated into a single design process. Such an integrated process was behind the success of the ALS optics program¹ and a similar pattern will probably be reproduced in other places.

These changes in the way beam-line optics are built happen gradually. The industrial infrastructures and manufacturing skills that are needed to implement the evolving designs take time to develop. At present only a few companies are able to build internally cooled synchrotron optics and indirect cooling continues to be a useful and available technique. However, it is certainly not the route to increasing ultimate performance levels and it is debatable whether it is a route to lower costs at moderate performance levels. Both of these R and D goals are best pursued in the view of this author via internal cooling schemes using less exotic materials than silicon carbide and with a "whole-system" approach to the design. The ceramic which is best suited to such an approach is silicon which is already replacing silicon carbide as the leading non-metallic candidate material and to this we now turn.

4.3. *Silicon*

Silicon has been a somewhat neglected material for beam-line optics in view of its excellent properties but that is now changing. Intrinsic single-crystal silicon has a thermal conductivity and expansion coefficient almost the same as silicon carbide and is readily obtained in large stress-free pieces of outstanding purity and uniformity made for the electronics industry. Moreover, there is a great deal of experience in the synchrotron-radiation community and elsewhere in cutting and shaping silicon for use in x-ray monochromators and interferometers including the use of designs with internal water cooling²¹. It is a matter of routine to fabricate internal cooling channels in silicon optics and most of the silicon mirror developments in recent years have involved much more elaborate structures. Nonetheless, published reports of the use of silicon for making mirrors are

rather few²², which is due in part to classification. It is known in the industry that first class figure and finish are obtainable in silicon but this is not yet well-documented in the open literature.

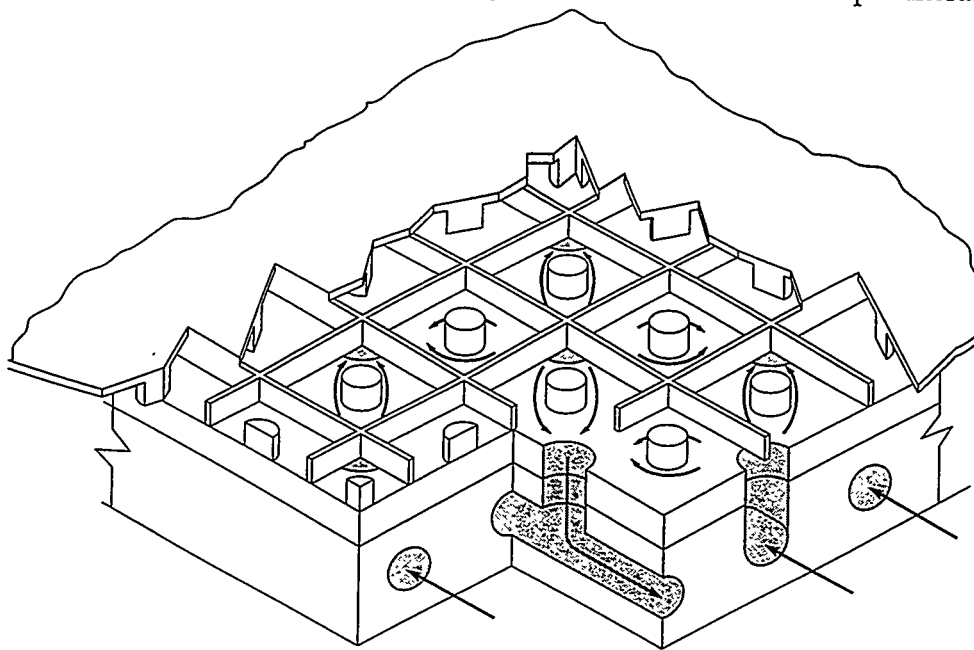


Fig. 3.

A number of major development programs for cooling high power laser mirrors were supported by the US government during the 1980's. These schemes were highly sophisticated and silicon was used in several of them. One, carried out by the group at Rockwell International²¹, produced a series of silicon mirrors cooled by water in the so-called "cellular-pin-post" geometry which is explained in Fig. 3. This scheme has been adapted for use on both beamline mirrors²³ and crystals²⁴. The construction consists of several silicon plates which are machined by conventional ultrasonic techniques and bonded together by means of melted glass ("frit" bonding). The design produces a rapid turbulent flow at the underside of the hot wall and heat transfer is by both the pin fins and the rectangular fins as well as directly to the underside of the wall. The key to the high performance is the flow geometry in which the water channels are narrow in the region where the coolant interacts with the hot wall and wider elsewhere. The effect is that for realistic pressures the coolant flow speed near the hot wall is as much as an order of magnitude higher than the values one can normally achieve with channels of uniform cross section. Since h varies roughly linearly with flow speed, one expects about an order of magnitude improvement in the heat transfer and this is indeed realized with overall heat transfer coefficients greater than $1 \text{ W/mm}^2/\text{°C}$ having been achieved. These manufacturing techniques hold great promise for use with synchrotron

radiation beam lines and several cellular-pin-post beam-line mirrors are at or near completion at the present time.

A different approach to cooling using silicon has been used by Tuckerman⁶. The work of this author on the cooling of silicon chips has shown that one can get very effective cooling with straight fins and channels on the order of 50 μm wide microfabricated in silicon. This moves the scheme into the laminar flow regime which has some attractions in mirror design because of "quietness" considerations. The Tuckerman designs gain an advantage in h value and maintain reasonable pressure drops by using sufficiently short flow distances. Overall heat transfer coefficients of the order of 0.1 W/mm²/°C were achieved for nominal 1cm flow paths. The same concept has been applied by Arthur and coworkers²¹ to cooling monochromator crystals on synchrotron-radiation x-ray beam lines and a similar h value was achieved. Microchannels are less appropriate for cooling large objects like grazing-incidence mirrors although they could still be considered when the beam footprint is sufficiently narrow.

4.4. *Metals in general*

Metal optics have been used since the earliest days of reflecting telescopes. For example, in 1778, the astronomer W. Herschel polished a 16-cm-diameter telescope objective mirror made from Molyneux's metal (71% copper, 29% tin). He used that telescope to discover Uranus and went on to try larger mirrors, some weighing hundreds of kilos, which were less successful. Nevertheless all telescope reflectors continued to be made of metal until around 1857 when an efficient process for silvering glass was discovered by Foucault²⁵. In modern times, nickel-plated metal mirrors, both cooled and uncooled, adaptive and rigid, particularly of aluminum and beryllium, continue to be used for telescopes. Now they are in the form of sophisticated grazing-incidence x-ray reflectors as well as the huge objectives of modern optical telescopes (see later).

Water-cooled metal mirrors²⁶ have long been used for applications involving high absorbed power densities, particularly from infra-red lasers, and are available commercially from a number of vendors. Electroless-nickel-plated copper and molybdenum have been the most popular materials with tungsten and aluminum also used. The figure accuracy of these infra-red mirrors is often based on the needs of carbon dioxide lasers ($\lambda/4$ or $\lambda/10$ say at $\lambda=10.6 \mu\text{m}$) and the finish is determined by the need for a high laser damage threshold (10-30Å rms is common). Thus, although they have some of the characteristics needed for high-power synchrotron-radiation beam-lines, the surfaces of standard commercial metal mirrors have not been of sufficient quality for modern requirements.

During the period around 1980, opticians tried to achieve the higher quality figure and finish needed for beam lines using nickel-plated metal substrates. For some time these attempts

were unsuccessful. Part of the reason was the low level of investment in optics by the synchrotron radiation community at that time. The technical problem, as pointed out by Becker¹⁷, was that the electroless-nickel material, and therefore its removal rate during polishing, was not sufficiently uniform. When the best quality finishing was attempted, it was also found to have insufficiently fine grain.

This situation has been dramatically turned around by recent developments in both the production and polishing of nickel-plated surfaces. Figure accuracies in the 0.5-5 μ radian range and finishes of 1-4 Å rms¹ (i. e. hardly any different than the best values for glass) are now being routinely achieved so that metal mirrors are now being made with both the accuracy and the cooling needed for the third generation light sources. Since these surfaces were worked in the plated nickel, the same success would apply to any substrate for which the nickel could be used. The properties of electroless nickel as an optical material have been reviewed by Killpatrick²⁷.

4.5. *Glidcop*^{TM28}

The Berkeley group have now made a number of glidcop optics for the Advanced Light Source beam lines which meet or exceed their optical specifications and are predicted by detailed ANSYS calculation to meet their thermal distortion specification. We regard the success of this program as well-established and well-documented. Apart from giving references, we do not consider it here since we are more interested in new possibilities. The key elements involved in making cooled optics of ALS quality are the thermal/mechanical engineering of the substrate^{29,30} electroless nickel plating³¹, optical working^{32,33}, ruling (in the case of gratings)³⁴, whole-surface metrology³⁵ and the integration of these into a real-world solution^{1,34,36}.

4.6. *Aluminum*

Aluminum is a convenient and inexpensive material and, with a nickel-plated layer for polishing, has been popular in varying degrees for making optics for high powered lasers, synchrotron beam lines and certain optical telescopes for the last thirty years or so. Generally, the optics have not been of the first quality figure and finish due to:

- Poor uniformity and grain of the nickel (as for other metal optics of the period),
- Thermal mismatch with the nickel and consequent bi-metallic bending,
- Dimensional instability of the gross figure of the substrate material of three possible types (i) metallurgical instability (ii) microcreep under load and (iii) stress relief.

Fortunately there has been considerable progress in recent years on most of these issues so that we can now consider aluminum for a much higher quality type of optic. Firstly, the problems of earlier

times with nickel quality have been successfully addressed as described earlier. Secondly, two solutions to the bimetallic bending have appeared: the "metal-matrix composite" SXA (see later), which can be thermally matched to nickel, has become available and also the technique of nickel plating *all* surfaces has evolved which reduces the bimetallic bending. Use of a stiff mirror shape also helps. Finally, there has been considerable research and development aimed at finding the most stable alloys and learning how to use them³⁷.

A large part of the effort to rehabilitate aluminum has come from the visible astronomy community and is driven by three main considerations

- Telescopes have grown too large for the traditional glass-based technologies to be cost-effective.
- Aluminum has about a hundred times larger thermal diffusivity than glass representing an approach to thermal equilibrium in minutes rather than hours.
- Adaptive designs have become necessary to hold figure during scanning of the sky so dimensional instability is less of a concern than hitherto.

A portion of the experiences with aluminum telescope mirrors have been with castable alloys such as A356 and tenzallo which would not be considered ultrahigh vacuum compatible and we discount these for beam-line applications. On the other hand there is also considerable experience with wrought alloys of which essentially three can be considered serious candidates: 6061-T6, the 5000 series (including 5083, 5086, 5456 and 5754) and SXA. The 5083 alloy is especially familiar to ALS engineers since it was used in making the giant ALS vacuum vessel.

The "optical grade" metal-matrix composite SXA^{38, 39} has a matrix of 2124-T6 (copper-containing) aluminum alloy and 30% of added fine-grain silicon carbide which leads to about a factor two higher elastic modulus and a factor two lower thermal expansion coefficient compared to standard aluminum alloys. The composition is chosen to give a thermal match to electroless nickel. This family of materials, which was developed to compete with beryllium for weight-critical applications, is machinable by conventional and electric-discharge machines and has improved dimensional-stability, microyield and creep properties compared to conventional aluminum alloys. An independent measurement of the dimensional stability of four samples of SXA, heat treated according to the manufacturer's specification, was made by S. Jacobs at the University of Arizona. It showed an average shrinkage of 5.7 ± 1.0 parts per million (ppm)/year. From an optical stability point of view, one should probably attach more significance to the ± 1.0 spread rather than the actual value of the shrinkage since the latter would not, on its own, lead to shape distortions. The chief disadvantage in using SXA will be the difficulty of welding and brazing. One solution is apparently to plate the surfaces with something like nickel and join them using tin-based solder.

On theoretical grounds, one can object to both 6061 and SXA because they are heat-treatable (i. e. thermodynamically-unstable) alloys whose properties depend on "aging" treatments.

However, such data as exist⁴⁰⁻⁴² on the shape stability of optics made from these three classes of alloy is not conclusive and good results have been achieved in certain cases with all three.

Table 1: Thermal cycling treatments for stabilization of aluminum alloy optics*

Author and material	Before finish machining	High temperature	Low temperature	Total # of cycles	Notes
[Vukobratovich 1993] ⁴¹ 6061-T6		191 for 3 hr	-40 for 0.5 hr	2	Rate <8°C/minute
[Vukobratovich 1993] ⁴¹ SXA	-59 for 1 hr	100 for 1 hr	-59 for 1 hr	5	Rate <3°C/minute
Average of several other sources	Anneal at 520**	185	Liquid nitrogen (-196°C)	5	Not specified
Manufacturer's treatment (SXA)	Anneal at 520**	177	Liquid nitrogen (-196°C)	several	Thought to be non-critical

* all temperatures in °C

**plus a quench and an aging treatment for 6061 and SXA or a slow cool for a 5000 series alloy

A major reason for the inconsistency of the published data on the dimensional stability of aluminum alloys is that there is no standard stabilizing heat treatment and some treatments are considered proprietary. In conventional engineering the recommended temperatures for heat treatment of the various aluminum alloys are 350-450°C for annealing and about 250°C for stress relief. On the other hand the literature of *optical* applications of such alloys shows a general practice of much more rigorous treatments including usually a solution anneal (at 520°C) after rough machining and then stabilization treatments consisting of thermal cycling between a moderately high temperature and liquid-nitrogen temperature. Such cycling is almost universally advocated⁴³ in spite of the effort involved and the fact that there seems to have been no systematic investigation of its effectiveness. One of the few authorities in this branch of engineering is D. Vukobratovich at the University of Arizona and we show in Table 1 his recommended treatments for alloys 6061-T6 and SXA⁴¹. We also show the manufacturer's recommendation for SXA and an average of the recommended treatments from several other sources which are generally described incompletely but are, in most cases, more rigorous than those of Vukobratovich. Some authors even recommend stabilization treatments at several stages during manufacture^{44, 45}.

Although only a few large telescope mirrors have been made of aluminum in the past (most of which were cast), aluminum alloys are now leading contenders for a number of important telescope projects. One is the 6 m diameter mirror which will be installed as the upgrade optic for

the 30-year-old McMath-Pierce telescope at the Kitt Peak National Observatory in Tucson, USA. Another is the Very Large Telescope (VLT) to be built at the European Southern Observatory (ESO) in Italy. In order to establish the best technical solutions for the VLT and other large telescopes, a project known as LAMA (Large Active Mirrors in Aluminum) was set up^{46, 47}. Two phases of studies and demonstration projects have been completed involving the manufacture of several mirrors of 0.5 m diameter for the first phase⁴⁸ and 1.8 m for the second⁴⁷. A great deal has been learned from which we may select the following items of interest for our application:

- Of the sixteen 0.5-m mirrors tested, seven showed good stability under thermal cycling (-20 to $+50^{\circ}\text{C}$), of which four were castable alloys and three wrought. Two of the latter were 1000 series ($>99.5\%$ pure Al) and one was a 5000 series (3% Mg).
- The 0.5-m figure stability measurements refer to high-spatial-frequency distortions. Simple focal length changes were not interesting to the investigators and were not reported.
- There was no correlation between the method of 0.5-m blank preparation (sand cast, open-mold cast, forged or rolled) and the observed stability of the mirror.
- The nearly-pure aluminum mirrors were expected to show better stability due to lack of heat-treatability effects but they did not do so.
- Response to thermal cycling was tested after completion of both types of mirrors and it appeared that the dimensions were often more stable after four cycles. This is evidence for the validity of using a stabilization sequence between nickel coating and polishing.
- The best 1.8-m blank was cycled several times between the annealing temperature and liquid nitrogen temperature before commencement of manufacturing.
- The figure of the best 1.8 m mirror was within 34 nm rms of the required 6.0-m-radius sphere (100 nm rms was specified) and figure changes due to 32 thermal cycles (-20 to $+40^{\circ}\text{C}$) were below 20 nm rms.

The evidence on choosing an aluminum alloy is by no means conclusive. The most-promising but least-known is SXA. A number of mirrors have been made from optical-grade SXA although none so far for synchrotron radiation. It should be competitive with glidcop for cooling of low to medium intensity and superior to it for large mirrors (especially toroids needing large waster plates) and benders where the specific stiffness and total weight are issues. This alloy appears to offer the best chance of bringing aluminum into the realm of first-quality synchrotron optics. For mirrors of lower cost it is still interesting to consider the question of 6061-T6 versus the 5000 series. The prevailing view in the literature favors the high-magnesium 5000 series, particularly 5083, for mirrors where a performance advance over and above the state of the art (of aluminum mirrors) is being sought. It is commonly available and has more favorable welding and diamond turning characteristics than 6061. We may cite its use for both of the LAMA 1.8-m blanks^{47, 49} and its advocacy by Taylor⁴⁵ and Franks⁵⁰. For mirrors within the state of the art of aluminum

optics the well-tried 6061-T6 has many supporters and will be hard to beat. It also brazes well which 5083 and most of the other 5000 series do not. 5050 is better for brazing and might be a compromise choice if brazing were high-priority. Whichever alloy is chosen, a critical ingredient of success will be careful application of the best stabilizing thermal treatments.

4.8. *Invar*

Invar has the best thermal-distortion performance in the temperature range 0-80°C of all the materials considered here plus wide availability and most of the engineering advantages of a steel-type alloy. Such advantages include the capability to be welded, brazed and machined without major deviations from the normal practices for dealing with steels^{51, 52}. These are very significant advantages but they can only be exploited if the major issue of dimensional stability can be addressed. We consider this in more detail below. The importance of invar has lead to a great deal of study and its length-change behavior for both varying and constant temperature has an extensive literature. Saito and McCain-and-Maringer^{53, 54} have summarized most of the work up to about twenty years ago while Jacobs, in whose laboratory many of the recent measurements have been made, has reviewed the more recent work⁵⁵.

The low expansion properties of the nickel-iron alloys were discovered by Charles Guillaume in 1886^{56, 57}. Invar (36%Ni+64%Fe) is the alloy with the lowest expansion coefficient in the neighborhood of room temperature. It is an austenitic (face-centered cubic) material which is ferromagnetic at room temperature with a Curie temperature of about 260°C. The low expansion property results from a balance between a decrease in atomic spacing associated with the loss of ferromagnetic ordering as the Curie temperature is approached and the normal increase in atomic spacing with temperature. For our purposes the practical range in which the expansion coefficient is low is about 0-80°C. At higher temperatures it rapidly reverts to a behavior similar to that of other steels. The invar property is compromised by certain impurities, particularly carbon, manganese and silicon. Expressed in ppm/°C/0.1% of impurity, the increase in the expansion coefficient is 0.4 for carbon and 0.15 for manganese. Impurity silicon does not affect α directly but reduces the useful temperature range of the invar property. The carbon content is thus of particular importance and should ideally be below about 0.01% but this is not normally achieved in commercial invars. Low values of α can also be produced by both heat treatment and cold working, although cold working would not be indicated for optical applications because its effects are neither permanent nor isotropic.

The main difficulty in using invar as a material for making mirror substrates is the fact that, without careful countermeasures, it changes its dimensions with time. This property has long been known and was studied by Guillaume who measured one sample at constant temperature for almost

30 years. The result of this and other studies was that commercial invar does stabilize within a ppm/year or so after a sufficient time but at room temperature, it may take many years. The principal effect is an expansion (known as the γ expansion) which when complete, amounts to a growth of about 50 ppm. An understanding of the γ expansion and the development of countermeasures to it were first achieved in a landmark study by Lement, Averbach and Cohen⁵⁸. These authors used x-ray analysis to show that the γ expansion is a true volume, i.e. lattice-parameter, change. They also found that there are three main effects to be concerned about in using invars (i) stress relief, (ii) the γ expansion and (iii) graphite formation. If the invar contains carbon above about 0.02%, as most commercial invars do, then its presence as graphite must be avoided or it will raise the coefficient of thermal expansion. Now the solubility of carbon in invar is 0.18% at 830°C and zero at room temperature. Thus the invar must be heated to 830°C to dissolve the carbon and then *quenched* in order to suppress the precipitation of graphite. The consequence of the quench is significant stress but this can be relieved, according to Lament et al., without precipitating graphite or raising the expansion coefficient provided the stress-relieving temperature is kept below about 315°C. After these procedures the material is still capable of undergoing the γ expansion, but results showed that it could be made to take place rapidly at an elevated temperature and could be fully completed in 48 hours at 95°C. An important related finding was that not only the graphite effect but also the γ expansion disappears for invars with carbon below about 0.02%.

The conclusion of the work of Lament et al. was thus the following three-step heat treatment for invar (the MIT triple treatment) that has received wide acceptance in the literature as providing the best combination of low α and good dimensional stability: after rough machining and high temperature procedures such as brazes

- (i) heat to 830°C for 30 minutes, water quench,
- (ii) heat to 315°C for 1 hour in air, air-cool,
- (iii) heat to 95°C in air for 48 hours, air cool to room temperature.

It is clear from the arguments presented, that the triple treatment is essentially a way to deal with the presence of unwanted carbon and is thus applicable to commercial invars. With its help one can get expansion coefficients near zero (0.15-0.4 ppm/°C for temperatures 0-80°C⁵⁹) and reasonable length stabilities.

The data on just how good the length stability can be after the triple treatment are sparse and somewhat contradictory. There is a general belief that the triple treatment favors good values of the expansion coefficient at the expense of dimensional stability and this has lead to a practice of replacing the quench in the first step with a slow cool or even of omitting the anneal altogether. Such procedures were used by Schwab and coworkers⁶⁰ who measured α values of 2-2.5 ppm/°C and an (isothermal) length change of 8-11 ppm/year with a standard invar at 38°C and an α value of 0.9-1.9 ppm/°C and a length change of 0.9-2.5 ppm/year with a low-carbon invar at 38°C. On the

other hand Marschall and Maringer reported a length change of 1-2 ppm/year following a full triple treatment including a quench. Other results (Table 23 in McCain and Maringer)⁵³ suggest that the first step of the triple treatment is not severely prejudicial to stability and that an aging step of a month at 70°C after the triple treatment reduces the length change rate to less than 5 ppm/year (these results were only quoted to the nearest 5).

A recent study by Steel and coworkers⁶¹ may resolve some of the apparent contradictions. These workers measured the length changes of high (0.06%) and low (0.02%) carbon invar after both the triple treatment and the triple treatment with the quench replaced by a slow cool. They found that the results could be represented by two exponential growth processes: a fast one of time constant 0.26 years with an initial rate of 10 ppm/ year and a slow one of time constant 3.0 years with an initial rate of 4.9 ppm/year. For the *slow* process, which is the most important one in practice, the length change rates were as shown in Table 2.

Table 2: Long-term length change rates of invar measured by Steel et al⁶¹

	Room temperature	Room temperature	38°C	38°C
	High carbon (ppm/year)	Low carbon (ppm/year)	High carbon (ppm/year)	Low carbon (ppm/year)
Full triple treatment	2.1-3.8	2	2.0-4.6	0.8
Triple treatment with a slow cool instead of a quench	2.3	0.7	2.9	0.5

Given that there is both a fast and a slow growth rate we at least have the possibility to reconcile all of the above results from the Schwab, McCain, Marschall and Steele references. The observed benefits of aging are confirmed and since the Schwab measurements lasted 47 days and the Steele ones 258 days the broad picture becomes reasonably consistent. Furthermore, all studies agree on the benefits of low carbon for both the expansion coefficient and the stability. The tentative conclusion is that commercial invars without special reduction of carbon can give expansion coefficients in the range 0-0.5 ppm/°C and stabilities of 2-4 ppm/year provided the triple treatment plus an aging step is used. If carbon is low 1-2 ppm/year may reasonably be expected.

So far we have concentrated on dealing with carbon-containing commercial invars, however, although invar alloys with carbon below 0.02% are not readily available, they can be obtained to special order. They should be free of both the γ expansion and graphite precipitation and thus offer another possible route to both low expansion and good stability. This has been the thrust of recent

developments by the Jet Propulsion Laboratory (JPL)⁶² for application of invar to thermal stabilization systems in spacecraft. The basic approach is to use powder metallurgy to achieve a highly controlled composition with carbon especially held below 0.01%. The triple treatment was again applied except that with such a low carbon level there is no longer a need for a quench following the initial high temperature anneal. The invars produced in this way have been tested by Jacobs' group and have shown expansion coefficients below 1 ppm/°C and stability better than 1 ppm/year using the triple treatment with the quench replaced by a slow cool. For comparison "standard" fused silica has $\alpha=0.6$ ppm/°C and a length change rate of 0.2 ppm/year.

These expansion and stability figures of the JPL invar are certainly within the range needed for high quality optics. Moreover, the JPL material has already been made in large enough pieces for beam-line mirrors. It thus appears that both commercial and JPL-type invar offer very interesting possibilities for beam-line mirrors on condition that the operating temperature is kept below 80°C.

5.0. Conclusions and ideas for further research

For the most challenging combinations of heat load and distortion specification where there is a need for very intensive (and thus internal) cooling, the present practice in the synchrotron-radiation community seems to be to use nickel-plated glidcop or silicon. For less severe challenges the same materials or silicon carbide are employed and cooling may be direct or indirect. For the mildest heat loads, fused silica or ULE are naturally still the most popular.

We are interested in how we can improve both the performance and the price in the future. For the highest performance mirrors, where the emphasis is on dealing with an extreme heat load we believe that the way forward is to continue the glidcop developments perhaps to cellular-pin-post systems. When the emphasis is on complying with extreme distortion specifications then it appears that silicon is indicated and that invar offers a promise of both improved performance and lower price. For less extreme challenges but still with cooling, it seems clear that nickel-plated metals have the cost advantage and that SXA and other aluminum alloys can be added to glidcop and invar as candidate materials. For mirrors with sufficiently mild cooling requirements, stainless steel would have many advantages including low cost and minimal technical risk. In the opinion of this author, once the internal cooling designs are established, they will be seen as more cost-effective and reliable than clamp-on schemes and the latter will gradually lose popularity. Continuing down the scale, there is a range of optics where no coolant is needed and radiation cooling can suffice. Silicon and silicon carbide are indicated here, in part due to emissivity considerations. Finally, for the range where no special cooling arrangements are to be made and the mirror is a simple rectangular block, the ceramics silicon, silicon carbide and the glasses have the advantage. From

this analysis it appears that, although silicon carbide offers certain capabilities, it is almost never the simplest and most cost effective solution to beam-line mirror needs.

For future research we identify the following as interesting items to pursue:

- apply the cellular-pin-post design to glidcop,
- study the long-term dimensional stability of the materials we use now as well as new candidates,
- include the effect of electroless nickel layers on cooling designs (it is often neglected at present),
- develop a way to finish nickel that is compatible with multilayers, i. e. achieve low roughness at all spatial periods down to near-atomic dimensions implying surface measurement by atomic force microscope as well as the optical profiler (Wyco or equivalent).

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