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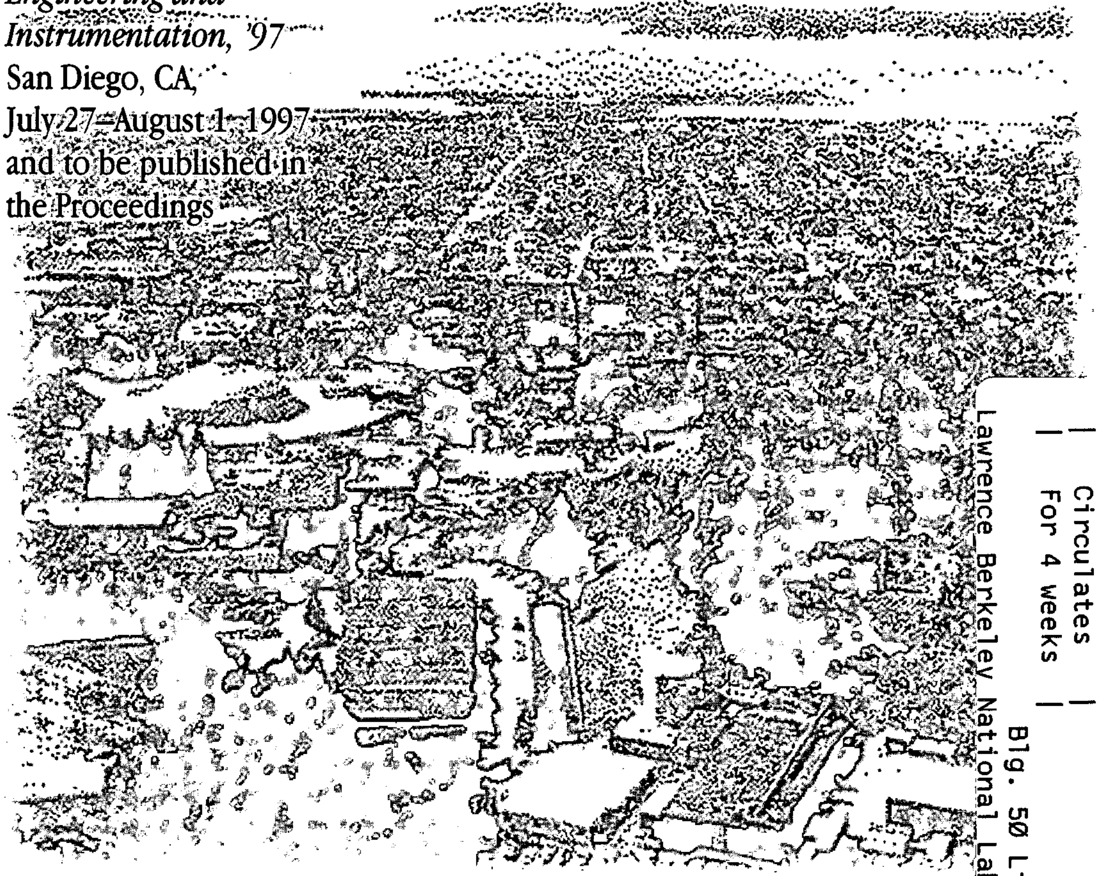

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Optical substrate materials for synchrotron radiation beamlines

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ABSTRACT

We consider the materials choices available for making optical substrates for synchrotron radiation beam lines. We find that currently the optical surfaces can only be polished to the required finish in fused silica and other glasses, silicon, CVD silicon carbide, electroless nickel and 17-4 PH stainless steel. Substrates must therefore be made of one of these materials or of a metal that can be coated with electroless nickel. In the context of material choices for mirrors we explore the issues of dimensional stability, polishing, bending, cooling, and manufacturing strategy. We conclude that metals are best from an engineering and cost standpoint while the ceramics are best from a polishing standpoint. We then give discussions of specific materials as follows: silicon carbide, silicon, electroless nickel, Glidcop™, aluminum, precipitation-hardening stainless steel, mild steel, invar and superinvar. Finally we summarize conclusions and propose ideas for further research.

Keywords: optics, materials, x-ray, silicon, silicon carbide, metals, sycnchrotron radiation, beam lines

1. INTRODUCTION

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 synchrotron-radiation beam line. The issues have to be evaluated in light 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 the mirror be designed and fabricated within 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?

In this paper we will discuss the most important candidate materials and their properties in the light of these questions.

The context for this exercise is the construction of a beam line costing, usually, in the range $1-5M (including optical and front-end systems but excluding the experimental station and the insertion device, if there is one). Within this cost structure one has to buy 2-6 optical substrates for a grating beam line or 1-3 for a crystal beam line. Thus one cannot devote study and research to each mirror at the level one could to an x-ray-telescope mirror, for example. This makes it all the more important to have guidelines in place to simplify the choices involved.

To approach the subject systematically we first try to make some broad statements about the technical requirements of synchrotron-radiation beam lines and identify the consequences for material selection in a general way. We then identify the types of material properties and property groups that are important and provide tables of values for the materials that we think are interesting candidates. We emphasize that this process applies to *substrate* materials. We assume that the substrate will eventually carry an optical coating which will determine the optical properties of the mirror but that lies outside the scope of this article.

The issues involved in designing and building an optic are complex but certain ones have a particularly profound effect. Among these we may mention manufacturing, polishing, cooling, bending and dimensional stability. We provide sections discussing these questions in light of the needs of synchrotron-radiation optical systems. Having set out the issues and the options, we finally turn to a discussion of the properties of individual materials with extensive references from which further information can be obtained. To gain an overview of issues that will become involved we initially present a series of tables. In Table 1 there is a breakdown of properties (with notations) that can influence the behavior of a substrate material, either during manufacture or service. In Table 2 we give a list of optical requirements and corresponding material properties, while in Tables 3 and 4 we provide values of mechanical and thermal properties respectively of a group of interesting or potentially interesting materials.

2. TECHNICAL REQUIREMENTS FOR SYNCHROTRON-RADIATION OPTICAL SUBSTRATES

We show in Table 2 an outline of the logic that leads from the technical requirements on a mirror to the material properties of the substrate material. The starting points are the optical specification of the mirror (the application) and the requirements of the environment (technical and economic). These are listed in a general way in the left column of Table 2. More detailed requirements mostly in the form of performance numbers that could be specified are listed in the second column of the table while the implications for material properties are indicated in the third column.

For a specular reflector, the shape of the optical surface will have a figure-error tolerance expressed as a rms slope and a roughness tolerance expressed as a rms height. At the present time typical high-quality optics have values in the ranges 0.3-3 microradians (μr) and 1-5 Å. We will define the meaning of the finish specification and

Table 1: Material properties influencing the behavior of an optical substrate

Mechanical	Physical	Microstructural	Fabrication-related
Young's modulus, E	Density, ρ	Crystal strucure(s)	Machinability
Yield strength, σ_Y	Thermal expansion, α	Phases present	Forgability
Microyield strength, $\sigma_{\mu Y}$	Thermal conductivity, k	Grain size	Platability
Creep strength	Specific heat C	Stress relief temperature	Polishability
Fracture toughness, K_{Ic}	Vapor pressure	Annealing temperature	Weldability
Modulus of rupture	Corrosion by air and Ga/In	Recrystallization temperature	Brazeability
		Softening temperature	Solderability
		Heat treatability	Machinability
		Density of voids & inclusions	
		Dimensional stability	

Table 2: Optical requirements, consequential requirements and material properties

Primary requirements	Consequential requirements	Material properties
Application-driven:		
Size		Size availability
Image quality	Figure spec.	Polishability Dimensional stability Adhesives: low shrinkage
Scattered light control	Finish spec. ripple Finish spec. roughness	Polishability: ceramic preferred over metal but ELN and 17-4 PH S/S also work
Multilayer compatibility	Finish spec. atomic scale	Ceramics only
Active bending	Strength spec.	Yield strength, microyield strength, creep strength, modulus of rupture, fracture toughness
	Mirror-to-bender connection	Metal preferred over ceramic Ceramic with adhesive also works
Environment-driven:		
Toleration of power load	Thermal distortion spec.	k/α (conduction-limited) $1/\alpha$ (convection-limited)
	Temperature spec.	k
	Speed of approach to equilibrium spec.	Thermal diffusivity
	Thermal stress spec.	Thermal stress FOM
	Thermal cycling spec.	Fatigue endurance limit
Toleration of gravity	Gravity distortion spec.	Specific stiffness
UHV compatibility	100% dense Bakeable to 100°C	Low vapor pressure: substrates and adhesives
	Clean surfaces	Low corrosion
Reasonable cost	Off-shelf materials preferred to near-net-shape approaches	Metals preferred if engineering is complicated
	Fabricability requirements	Brazeability, solderability, machinability, weldability
	Flat-polishing-plus-bending preferred to aspheric figuring	See properties under "active bending"

ELN=electroless nickel, S/S=stainless steel, FOM=figure of merit

Table 3: Mechanical properties

	State of heat treatment	Density (ρ) gm/cc	Young's modulus (E) GPa	Poisson's ratio	0.2% yield stress MPa	Microyield stress MPa	Fracture toughness (K_{Ic}) MPa$\sqrt{m}$	Modulus of rupture MPa
Fused silica		2.19	73.00	0.17			<1.00	110.00
ULE fused silica		2.21	67.00	0.17			1.00	
Zerodur		2.53	92.00	0.24			1.50	
Silicon: single crystal		2.33	131.00	0.42			1.00	207.00
SiC, CVD		3.21	461.00	0.21			2.70	595.00
SiC, RB 30% silicon		2.91	413.00	0.24			2.50	290.00
Electroless nickel	unannealed	8.00	110.00	0.41				
Electrolytic nickel		8.90	199.50	0.3	148.00			
Aluminum: 6061	T6	2.70	68.00	0.33	276.00	240.00	18.00	414.00
Aluminum: 5083	O	2.66	71.00	0.33	145.00	40.00	27.00	
Aluminum: SXA™	T6	2.91	117.00	0.29		>200.00	>10.00	
Copper: OFHC	Fully annealed	8.94	117.00	0.343	195.00	12.00		
Copper: Glidcop™	Fully annealed	8.84	130.00	0.33	331.00			
Molybdenum		10.22	324.80	0.293	600.00	280.00		
Beryllium:I-70		1.85	287.00	0.043	276.00	30.00	11.00	241.00
Invar 36	Triple treatment	8.05	141.00	0.259	276.00	70.00		
Superinvar	Triple treatment	8.13	148.00	0.26		75.00		
Steel: 304 stainless		8.00	193.00	0.27	241.00	>300.00		
Steel: 1010 mild	Fully annealed	7.86	200.00	0.28	180.00			
Steel: 17-4 PH stainless	H900	7.80	200.00	0.272	1170.00	>700.00	53.00	

Table 4: Thermal properties

	State of heat treatment	Thermal expansion (α) ppm/°C	Thermal conductivity (K) W/m/°C	Specific heat (C) J/gm/°C	Distortion figure of merit (K/α) µm/W
Fused silica		0.50	1.40	0.75	2.80
ULE fused silica		0.03	1.30	0.77	43.33
Zerodur		0.05	1.60	0.76	32.00
Silicon: single crystal		2.60	156	0.71	60.00
SiC, CVD		2.40	198.00	0.73	82.50
SiC, RB 30% silicon		2.50	155.00	0.67	62.00
Electroless nickel (11% P)	unannealed	12.50	7.40	0.46	0.59
Electrolytic nickel		13.40	70.00	0.46	5.22
Aluminum: 6061	T6	22.50	167.00	0.90	7.42
Aluminum: 5083	O	22.60	120.00	0.90	5.31
Aluminum: SXA™	T6	12.40	123.00	0.87	9.92
Copper: OFHC	Fully annealed	16.50	391.00	0.38	23.70
Copper: Glidcop™	Fully annealed	16.60	365.00	0.38	21.99
Molybdenum		4.80	142.00	0.28	29.58
Beryllium:I-70		11.30	216.00	1.92	19.12
Invar 36	Triple treatment	0.50	10.40	0.52	20.80
Superinvar	Triple treatment	0.05	10.50	0.51	210.00
Steel: 304 stainless		14.70	16.20	0.50	1.10
Steel: 1010 mild	Fully annealed	12.20	60.00	0.45	4.92
Steel: 17-4 PH stainless	H900	10.40	22.20	0.46	2.13

discuss it further in section 4. However, the need for a good finish has a dramatic influence on the question of material choices because only a fairly small number of materials can be polished to a finish of <5 Å rms. The best-known of these are the glasses, particularly fused silica, ULE™ fused silica[15] and Zerodur™[72]. A second group consists of single-crystal silicon and chemical-vapor-deposited (CVD) silicon carbide. Finally there are electroless nickel (ELN) and 17-4 PH stainless steel. There are also several materials which have been polished to finishes which lie just outside the "superpolish" range, such as invar, reaction-bonded silicon carbide and molybdenum and we may regard these as future contenders. Of the core group, 17-4 PH stainless steel, although a very-well-known material, is a newcomer to the realm of superpolishing. We discuss its possible future role in section 8.7. Since ELN can be plated on to most metallic substrates, its use allows them all to be included in the range of substrate choices. However, the use of optical-grade ELN is not without effort and cost and it complicates the manufacturing process. Moreover it cannot yet be polished to an adequate finish to work as a multilayer substrate. If the mirror is to carry a multilayer coating, then the finish requirement is more severe and extends to atomic scales. In this case only glasses and silicon have so far been successfully used.

A second major factor in synchrotron-radiation mirrors is bending[30] (section 5). This introduces a range of new materials issues which are mostly mechanical in nature. Historically, most bent mirrors have been uncooled. However, there are definite needs for first mirrors (receiving full beam power) that would be best made by bending. An example is a bending-magnet horizontal-collection mirror. If the desire is to collect the largest-possible angle then an elliptical cylinder close to the source would be indicated which would usually imply both cooling and bending. The largest group of mirrors made by bending in the past have been relay mirrors either transferring light from source to entrance slit or exit slit to sample. Such mirrors are not resolution-determining and so a lower level of optical quality could usually be tolerated. However, a significant number of mirrors have now been made at 1-3 μr rms figure accuracy by bending techniques[58] and it appears that the time may be right to consider tunable-radius mirrors or even gratings inside the monochromator. Success in this venture would carry a valuable payoff.

A third key issue is cooling. If the heat load is severe then a material with high conductivity is needed which could be Glidcop™ or aluminum with an ELN coating or it could be silicon or silicon carbide. To engineer a good cooling geometry with internal cooling channels is relatively difficult (i. e. expensive) with ceramics and generally more straightforward with metals. On the other hand the ceramics have the advantage in polishing. For milder heat loads, a wider range of materials can be considered and a class of designs involving indirect cooling via clamp-on plates becomes feasible. We will discuss this further in section 7. This general division between metals that are easier to engineer and more difficult to polish and ceramics which are the opposite is the essence of the materials problem in this field.

Since we normally have to make a large number of different optics within a fairly small budget, it is normally more suitable to use processes based on machining of off-the-self materials, preferably ones that are made in high tonnage quantities. Processes involving casting or sintering tend to have high initial tooling costs and are also suspect from a UHV-compatibility point of view. Similarly it is often cost-effective to make optics by bending substrates that have been polished flat so as to avoid aspheric polishing or even spherical polishing at very large sizes. The need to cut costs is a factor which points us in certain technical directions. It favors methods and materials that allow standard machine shops to make optical substrates which, in turn, favors the use of conventional engineering materials such as steel, aluminum and metals generally, especially where engineering of cooling channels is required. Among the ceramics, silicon is by far the most cost-effective for optical engineering operations. Another way to cut costs is to look for optical schemes which make repeated use of the *same* mirror designs. If this scenario were to become a reality then the technical logic would point toward mass-production approaches like casting of materials such as reaction-bonded silicon carbide and nickel electroforming. However, we are years of research and development away from that at present.

3. DIMENSIONAL STABILITY

In order to fabricate a useful optic with submicroradian surface accuracy, it is obviously necessary to have a substrate material which is stable with submicroradian accuracy. We are thus lead to enquire into the factors determining the dimensional stability of materials and to study ways to obtain optical substrates of sufficient stability which also satisfy the other requirements for beam-line optics. The starting point for such studies is the literature on materials for precision mechanical instruments which reflect research efforts going back about 50 years. These efforts, especially those at MIT, have been described by Lement[42] and Maringer[46] and more recent work is reported in the book by Marschall and Maringer[47] which is still the principal reference of the field. We also have the extensive literature generated by the program at The National Bureau of Standards (NBS) on the design of gage blocks[47, 51-54]. Interest in materials for advanced optics has developed more recently out of applications in high-power lasers, ground- and space-based astronomy and military systems[61, 62, 86]. Valuable information can also be found in the handbooks produced by ASM International[87]. Much of this material has been developed for applications that differ considerably from synchrotron beam-line optics. The technical solutions in the literature therefore provide only general principles which we will try to apply judiciously to the cases of interest.

3.1. Dimensional Instability Types

According to Paquin[60] the important types of dimensional instability can be classified into four groups. We list them below and in the following section we indicate the issues of interest in designing beam-line optics.

Temporal instabilities are dimensional changes that happen spontaneously without any corresponding change in the environment of the component. They may be thermally activated but they occur in service under constant-temperature conditions.

Thermal mechanical cycle instabilities imply a permanent dimensional change due to a *process.* It includes stress-altering treatments in manufacture (heat treatment) or in service (bakeout), microcreep under repeated bending loads and microstructural changes due to radiation. Often the dimensional change diminishes as the number of repeats of the process increases which is the basis for cycling treatments.

Thermal instability is a failure to make the dimensions of the optic independent of environmental variables although the dimensions are still simple functions of the variables without any memory effect. The important variable is temperature and the dimensional changes are associated with *nonuniformities* of either the system thermal expansion coefficient (thermal mismatches) or of the temperature distribution produced by the x-ray beam power load. To achieve temperature independence the strategy is to have good cooling (see section 6) and to build components from thermally-matched materials as far as possible. The desire to match the expansion coefficient of ELN was one of the motivations for developing optical-grade SiC-reinforced aluminum alloys.

Hysteresis is the same as thermal instability except that a memory effect is involved. We are not aware of any occurrence on a beam line where this has been significant.

3.2. Sources of Dimensional Instability

The instabilities described above arise from three primary sources: (i) applied external forces as in bending to shape (ii) changes in residual stress and (iii) microstructural change as a result of environmental influences. We give examples of these in the following discussion. We turn first to temporal instability which was important in the NBS gage-block project and is also high on the list of issues for beam-line optics.

The transformation of retained austenite to martensite or lower bainite is a thermally activated microstructure change that is among the processes that take place when a quenched martensitic steel alloy is tempered. Dimensional stability problems arise when the same process takes place gradually at the service temperature. The conventional countermeasure is to use low-temperature treatments to make the transition to martensite as complete as possible. The part is cooled slowly to about -60°C, held for few hours and then returned slowly to room temperature. This may be repeated 1-3 times.

Carbide precipitation and other carbon rearrangements are also temporal instabilities that are thermally activated microstructure changes occurring at constant temperature. Carbon plays a vital role in the dimensional stability of steels because it is an *interstitial* alloying element producing a much larger lattice distortion then the other common alloying elements which (apart from nitrogen) are substitutional. Roughly speaking, the specific volume of steel alloys changes by a huge one per cent for each one per cent change in carbon concentration. Thus when martensite-tempering-type reactions involving the movement of carbon (segregation to defects, precipitation of transition carbides and cementite etc.) occur, either rapidly at the tempering temperature or slowly at the service temperature, the corresponding dimensional changes are substantial. Carbide precipitation was the cause of the small instabilities of old-style through-hardened gage blocks, made from high carbon steel type 52100[54], that originally created the demand for the NBS gage-block project. More subtle carbon rearrangements are responsible for the celebrated gamma expansion in quenched invar. (See section 8.9). This is a total length change of about 50 parts per million (ppm) with an initial rate of more than 5 ppm/year that takes many years to complete at room temperature[43]. All of this suggests that one of the main strategies for achieving good dimensional stability in steels is to use alloys with very low carbon.

Alteration of residual stress is the biggest single cause of dimensional instability and can cause temporal as well as thermal/mechanical cycling instabilities[47, 60]. Internal stress can exist on a macroscopic scale, typically produced by manufacturing processes such as heat treatments or machining, or it can exist on a microscopic (grain-size) scale in which case it would be caused by thermal expansion anisotropies among neighboring grains. Either way, to preserve equilibrium, there must be a pattern of balanced tensile and compressive stresses. As a typical way to acquire macrostresses consider a cylindrical part undergoing a quench. The outer surface region cools first and experiences high tensile stress as it tries to contract against the, as yet, uncooled inner region. As a result the outer region yields leaving it in compression after the whole part has cooled. This example shows how the size of internal stresses can often amount to a substantial fraction of the yield stress. Conventional grinding, and milling with a sharp cutter, also produce similarly large compressive stresses over a surface region several tens of microns deep.

The dimensional changes due to residual stress are a form of creep and, at constant temperature, the rate of dimensional change will be determined by the size of the stored stress and the creep strength of the material. As the material moves, the stress relaxes so both the stress and the rate of movement show an exponential decay with time. Stress relief is a thermally-activated process so its rate is mainly determined by the temperature. This leads to the notion of stress-relieving heat treatments which we outline in the next section.

3.3. Some general strategies for achieving dimensional stability

The following practical countermeasures may be used against residual stress[47, 60].

(i) Use materials with high microyield stress and creep strength that will tend to tolerate the internal stresses without yielding. The gage-block test results probably provide a short cut to this.

(ii) Avoid deposited layers such as ELN or CVD SiC or design them for low stress.

(iii) Just before optical working, apply the most aggressive possible heat treatment, such as a full anneal, with long heating and cooling times (say 2 hours per centimeter of section).

(iv) Thermally cycle the parts a few times over a wider temperature range than they will see in service before final polishing to an optical figure. (See section on aluminum alloys.)

(v) If the part is to be used under stress then stress cycle it similarly.

(vi) If aggressive heat treatments are not practical, then consider mechanical vibration or a combination of mechanical vibration with mild heat treatment.

(vii) If stressed layers are known to be present at the surface, for example due to surface grinding[47], remove them using a low-stress process such as lapping or chemical etching.

The question of whether to use a single isothermal treatment (iii) or a cycling treatment (iv) is a difficult one. In theory[60] the cycling treatments should work best where there is a significant microstress component. This would be expected in non-cubic materials with reasonably high expansion anisotropy and multiphase materials such as fiber- or powder-reinforced composites. This expectation is born out by experiments but in the case of homogeneous-cubic or amorphous materials the evidence in the literature is conflicting.

It is always risky to bake a finished optic, even to the 80-100°C level that is commonly used. An alternative approach is (i) insist on good cleaning procedures and protection of holes etc from polishing slurry, (ii) bake the mirror box hard without the mirror in and then expose it only to dry nitrogen, and (iii) outgas the mirror with UV light. This procedure normally allows a pressure in the UHV range to be achieved. As an example of a distortion problem caused by bakeout we can cite the case of an ALS Glidcop™ refocusing mirror. The distortion resulted from the relaxation of internal stress because the mirror, being uncooled, had never undergone any brazing cycles which would normally have provided sufficient stress relief.

Dimensional stability under load is important in optics whose shape is formed by bending. The required material properties are high microyield stress and high creep strength both of which involve resistance to microyielding which is a process that initiates by the movement of dislocations. We might expect therefore that materials in which the movement of dislocations is inhibited by dispersion strengthening (Glidcop™ for example) or precipitation hardening (17-4 PH stainless steel or 6061 aluminum for example) would have good dimensional stability under load. All three of these materials do indeed have high microyield strength. It is often difficult to find information on microyield strength but the following is a general guide. For most classes of materials with similar thermomechanical history, the ratio of microyield stress to 0.2%-yield stress is constant. For example for heat-treatable aluminum alloys, the ratio is about 0.6. For non-heat-treatable or fully annealed alloys it is considerably lower, around 0.3-0.4. On the other hand for most beryllium materials the ratio is only about 0.15. Generally small grain, highly-alloyed or multiphase materials have the highest ratios.

So far we have mostly discussed dimensional stability in metals. This is because it is in relation to metals that dimensional stability questions normally arise. The traditional glassy optical substrate materials, under low stress and power loading, have well established and well measured stability[34]. Thus, under the conditions in which we would be most likely to choose a glassy material, typically a low-power refocusing mirror receiving monochromatic radiation, the stability will not be a concern. Similarly silicon and SiC are essentially free of temporal instabilities. Thus at low power and stress, the stability of a ceramic substrate can usually be assumed while that of a metal one has to be achieved by careful design. However, this simple situation is beginning to change. Since glass and silicon are the only materials that can be used for multilayer substrates, they may be called upon to operate under stress, which means we must now begin to study their microyield and creep properties and the mechanisms which cause them to fracture[55]. For cooled mirrors, the temperature will usually not be constant due to the exponential decay of the stored electron-beam current, and then thermal stability must be added to temporal stability as a condition for a stable optical surface.

4. POLISHING

The two simple numbers traditionally used to specify figure and finish; the rms slope error and rms roughness height, fall far short of a full definition of the surface scattering properties. A complete definition would require the prescription of an upper bound on the surface power spectral density (PSD) function at all spatial frequencies from atomic-scale to mirror-size-scale. However, although our understanding of the theory allows us to calculate the correct upper bound to guarantee the desired performance, opticians cannot, in general, build power spectra to order. Attempts to understand polishing processes well enough to do this are a subject of active research. The standard procedure to buy a synchrotron-radiation optic at present is to specify a slope-error tolerance which will be verified by a long-trace profiler[79] (frequencies of (mirror-length)$^{-1}$ to 1 mm^{-1}) and a roughness tolerance which will be checked with an optical profiler[85] (frequencies 0.3-100 mm^{-1} with a 2.5× objective). If the optic is within the tolerances *and* has been made with a large tool (see below), then the power spectrum seems to be generally satisfactory for grazing incidence specular mirrors doing the job from which the slope-error tolerance was derived.

The best results are usually obtained with optical tools which are large compared to the work piece and in 100% contact with it all the time. This is only possible for flats, spheres and circular cylinders. The use of smaller tools ("zone polishing"), which is necessary to polish more difficult shapes, usually leads to errors with a spatial scale similar to the tool size. This fact is an important argument for limiting the optical surfaces used in beam lines to flats, spheres and circular cylinders or shapes that can be formed from those by bending.

The experience of the Berkeley group in procuring optics goes one step further than favoring a large lap. We have had the best results with laps which are designed so that the shape of the pitch surface is not controlled by its interaction with the workpiece but, rather, with a special, permanently resident conditioning weight. Such a system, also known as a planetary lap or continuous polisher, can satisfy the need for a large tool and also seems to allow more independent control of figure and finish. It was with this type of tool that the surface roughness of ELN-plated Glidcop™ substrates (measured as above) was first reduced down to the 2Å level[44]. Moreover, figure errors in the same optics were also reduced to a level (0.3-0.6 μr) that enabled soft-x-ray spectra at the unprecedented resolving power of 65000. See sections 8.4 and 8.5 for further information.

5. BENDING

Making mirrors by bending an initially flat or cylindrical substrate has a long history (see references in Howells and Lunt 1993[30]) and is now applied to both astronomical optics[41] and synchrotron-radiation ones and to complex non-circular shapes such as elliptical cylinders[58] as well as to circular cylinders. The first requirement of bending is that the mirror be joined to the bending machine in some way. This is easy for metals, one can simply use nuts and bolts, but for the ceramics it usually implies the use of adhesives or special solders, both of which raise questions of ultrahigh-vacuum (UHV) compatibility and of shrinkage (with possibility for mirror distortion). Our experience has been that in both of these cases it is important that the applied forces (due to bolts or shrinkage) be directed *perpendicular* to the mirror surface. This is understandable in light of the fact that the stresses that produce bending of a beam are *longitudinal.* Bending also introduces issues of brittle fracture for the ceramics and creep for all of the materials. The most difficult challenge arises when severe bending is required, say to radii below 10 m. In this case the tolerable stress level for the ceramics requires that the thickness be reduced to around 1-2 mm which gives insufficient rigidity for high-quality polishing. For metals, it is easy to find materials with a high stress capability, which allows thicker, and thus more polishable substrates. However, they do not offer a path to multilayer-coated mirrors nor is it demonstrated that an ELN coating can perform satisfactorily under high bending stress. This was the motivation for developing the superpolishing of stainless steel (see section on 17-4 PH stainless steel). The success of that program now allows us to build small mirrors out of steel alone (so far to 1-3 μr figure accuracy) which is a great simplification. The next steps will be toward larger sizes and the introduction of cooling. Stainless steels have a thermal conductivity which is intermediate between that of the glasses and the best conductors, so there is a range of heat loads for which they provide the simplest solution.

6. COOLING

The design of cooled mirrors for synchrotron-radiation beam lines is already a growing branch of optical engineering with a substantial literature including a number of reviews[5] workshop reports[21, 74], conference proceedings[36, 37] and a special issue of Optical Engineering[38]. The basic principles involved are covered in the standard texts on heat transfer[12, 28, 35] and there are other well-developed technologies with similar requirements, the closest being high-power-laser mirrors (especially continuous-wave ones[59]).

Our purpose here is to consider the effect of the x-ray beam power load and the consequent cooling strategies on the choice of materials. First, it can be shown[29] that under fairly general conditions the temperature rise, for a given heat load and cooling geometry, varies like $1/k$. This has a simple consequence for dealing with extreme heat loads where the first priority is to ensure stable cooling and the survival of the optic by keeping its temperature safely below the boiling point of the coolant. In this case, the conductivity becomes the dominant consideration so that copper and Glidcop™ are strongly favored while Al, Be, SiC, Mo and Si have conductivities about a factor 2-3 worse. Even for quite moderate heat loads, temperature considerations tend to rule out glasses because of the high temperatures consequent upon their poor conductivity. (They would be unattractive anyway on engineering grounds.) Overall there are good reasons, based on dimensional stability and stress arguments, for keeping the temperature of an optic as low as possible, even when that is not mandated by distortion considerations. In the case of low-expansion materials, a low temperature is doubly important because the low-expansion property only applies over quite a narrow temperature range (see Table 4).

Another conclusion that we can draw from a general analysis[29] is that the heat removal from a mirror has two aspects: conductive and convective, whose relative importance is represented by their respective thermal resistances. For good conductors the convective thermal resistance tends to dominate and we speak of convection-limited heat flow. Conversely, poor conductors tend to produce conduction-limited heat flow. Now the contributions to the thermal distortion are proportional to α/k for conduction-limited flow and α/h for convection limited flow, where h is the convective heat-transfer coefficient (measured in W/mm^2/°C). This has given rise to the assumption that the ratio α/k is the principal consideration in choosing a material for low thermal distortion. However, this is only true for conduction-limited flow and, in practice, cooled mirrors are often made from good conductors and the heat flow is often convection-limited. In such cases the material figure of merit is $1/\alpha$ since h is not a material property. Either way it is clear that invar and other low expansion materials such as silicon, silicon carbide and molybdenum have to be given special consideration in choosing materials for cooled optics.

The general strategies in designing a good mirror cooling system are as follows;

- Reduce the conductive thermal resistance by having a sufficiently short conduction path between the mirror surface and the coolant.
- Reduce the convective thermal resistance by having a high coolant contact area and flow speed.

The short conduction path (l, say) is important because the distortion (Δl) tends to increase like the square of l. ($\Delta l \propto l\alpha\Delta T$ and $\Delta T \propto l$).

7. MANUFACTURE

7.1. Directly cooled mirrors

A simple and common type of cooled mirror involves a face plate in which cooling channels have been machined and a rigid substrate to which the face plate must be joined. Such a joint may be made by braze, solder, melted glass ("frit"), diffusion and perhaps other methods. The two common choices are frit bonding for silicon and brazing for Glidcop™. For intense cooling it is important to keep the hot wall thin, 1-2 mm typically. However, for less demanding cases, where the wall can be thicker, gun drilling may provide an even simpler design without the face-plate-to-substrate joint. However, in this case, as in all the designs, there is still the need to provide coolant connections, usually with a requirement for no vacuum-to-water joints. This generates a need for joints or seals between the plumbing and substrate materials. A similar design problem must be solved for the cooling plates in indirect-cooling designs. In all these cases, the necessity to make the required joints with appropriate leak-tightness and vacuum protection places important limitations on the choice of materials and highlights the engineering advantages of working with metals. For both direct and indirect designs, a simplification may sometimes be made if the cooled object is made part of the vacuum wall. The coolant can then be connected outside the vacuum which alleviates some of the difficulties.

7.2. Indirectly cooled mirrors

Another approach to cooling mirrors is indirect cooling of the mirror sides via cooled pressure plates. This method has gained considerable popularity in the synchrotron-radiation community for mirrors where the most intensive cooling is not required. Although this type of scheme has played a useful role in the evolution of the field, we believe that in the future the trend will be toward more direct-cooling schemes. These will be needed for the more difficult heat loads and more challenging optical-surface-accuracy specifications of future generations of sources and beam lines.

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 connecting 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. For the highest-heat-flux devices (currently radio-frequency amplifiers and laser diodes) water-cooling channels have been integrated into the device package[84] while for the Pentium computer chip internal air-cooling channels have been included. It would appear that the synchrotron optics technology is in the middle of a similar sequence of developments where 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 as close as possible to the applied heat load. This does not necessarily drive up the cost of the whole system but it leads to different designs and favors metal and silicon mirrors that simplify the engineering of internal cooling. Moreover, it requires that the optical, mechanical and thermal aspects of the project be integrated into a single design process.

8. INDIVIDUAL MATERIALS

8.1. Silicon carbide

Recognition of the x-ray-beam-power problem in the 1970's lead to the notion of silicon carbide mirrors[68] which had originally been proposed in 1976 by Choyke[13]. 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 alpha 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. This material is inexpensive and 100% dense and is manufactured on a large scale, for example by British Nuclear Fuels[10] as Refel and Carborundum Company[11] as KT. Moreover, it can be machined and then cast to near net shape[80]. The material has about a 10-30% excess of silicon which is non-uniformly distributed and this has so far prevented the raw material from being used directly to make superpolished optical surfaces[45]. The material which *can* be superpolished is the chemical-vapor-deposited (CVD) beta silicon carbide which can be coated on a suitable substrate by pyrolysis of methyltrichlorosilane in an excess of hydrogen in a low-pressure CVD reactor[22]. The rate of deposition is slow, generally less than 0.1mm/hour, so thick coatings are difficult and expensive. They are also prone to high stresses. The group at Morton International[57] have produced some of the largest pieces of optical-grade silicon carbide and have made extensive measurements to characterize the material[23, 24, 67].

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 graphite[71]. 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 a few laboratories make specially for each application. Its physical properties are sensitive to many production parameters[6, 24] and are not yet repeatably measured. Mirrors consisting of a laminate of two different polytypes show detectable thermal instability[45] although the individual polytypes are essentially free of temporal instabilities. The extreme hardness of silicon carbide is a major disadvantage in working the material which becomes limited to specialists. Even more serious is the fact that internal cooling of CVD-coated-substrates is so difficult that, although some attempts are currently in progress, an internally-cooled synchrotron-radiation optic of this type has still not been built. The silicon-carbide technology has therefore not been applied to the most challenging thermal problems which were 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. In summary, silicon carbide is currently limited to uncooled or indirectly-cooled mirrors comprising a simple rectangular block of material and this is a manufacturing problem which is much more easily solved in other ways. Silicon, in particular has equally good thermal properties and to this we now turn.

8.2. Silicon

Until about the time of the construction of the European Synchrotron Radiation Facility beam lines in the early 1990's, silicon was a somewhat neglected material for beam-line optics. Now silicon beam-line mirrors are almost as widely available as glass ones. Intrinsic single-crystal silicon has almost all of the advantageous properties of silicon carbide without the disadvantages. It is manufactured in tonnage quantities in large stress-free pieces of outstanding purity and uniformity 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 other x-ray optical devices including the use of designs with internal water cooling[4, 84] Bragg optical devices involving bending, including bending to extreme curvatures for sagittal focusing, have been quite widely used. The rule of thumb in such cases is that for a crystal that is not chipped, a bending radius of 1000 times the thickness can be achieved[20]. One would expect a single-crystal-material to be dimensionally stable and the successful use of silicon crystals for Bonse-Hart x-ray interferometry experiments certainly demonstrates a level of stability in at least the 10 nanoradian range which is well beyond the needs of mirror substrates. As an optical engineering material[7, 66] silicon has been known for some time but the literature on it is limited, in part, by classification. The historical review by Anthony[3] provides good documentation of available literature.

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 International (now Boeing North American)[9], produced a series of silicon mirrors cooled by water in the so-called "cellular-pin-post" geometry which is highly efficient. The construction consists of several silicon plates which are machined by ultrasonic techniques and frit bonded together. The design produces a rapid turbulent flow at the underside of the hot wall and heat transfer is by both pin fins and 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 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. This indeed produces about an order of magnitude improvement in the heat transfer coefficient.

The pin-post scheme can be used for both beam-line mirrors[81] and crystals[82] and the technique of frit bonding of machined silicon plates can also be applied to a wider class of geometries including simple uniform cooling channels. At the time of writing about half a dozen mirrors and a dozen crystals involving both types of cooling have been made by Boeing for synchrotron-radiation applications. The mirrors have included both large size and (1m) and high quality (2 μr) surfaces and this approach to making cooled beam-line optics is now established as one of the successful routes.

8.3. 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 Foucault[19]. In modern times, ELN-plated metal mirrors, 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.

Water-cooled metal mirrors 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. Copper, molybdenum, tungsten and aluminum have all been used both with and without ELN coatings. These mirrors have some of the characteristics needed for high-power synchrotron-radiation beam-lines, but, due their infrared-oriented specification, they are a little below the needed optical quality. During the period around 1980, opticians tried to achieve the higher quality figure and finish needed for beam lines using ELN-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 Becker[6], was that the

ELN material, and therefore its removal rate during polishing, was not sufficiently uniform. This situation has been dramatically turned around by recent developments in both the production and polishing of nickel-plated surfaces. Figure accuracies in the 0.3-3 μradian range and finishes of 1-4 Å rms[14, 49] (i. e. hardly any different than good values for glass) are now being routinely achieved so that metal mirrors are being made with both the accuracy and the cooling needed for the third-generation synchrotron light sources. Since these surfaces were worked in the plated nickel, the same capability should apply to any substrate for which the nickel could be used.

8.4. Electroless nickel

Electroless-nickel (ELN) coatings[27, 39] are used on beam-line optics to provide a layer that *can* be superpolished on substrates that, otherwise, could not be. It is an alloy of nickel and phosphorus which, for optimum pitch-polishing, needs to be hard and amorphous. One condition for obtaining these two qualities[78] is that the weight % of phosphorus should be in the range 9–11. The procedure for deposition would then be as follows.

(i) Clean and activate the substrate surface to initiate the autocatalytic reaction.

(ii) Plate the surface. This is usually done with proprietary solutions that contain nickel and phosphorus ions and a reducing agent (often sodium hypophosphite) as well as additives to control the subtleties of the process. The important variables are the temperature and pH of the bath (normally kept around 90°C and 4–5 respectively) and the ion concentrations. By monitoring and adjusting these variables, the plater can control the percentage of phosphorous in the coating. Usually, a coating thickness of about 75–125 μm is applied over a 10-40-hour period, which is intended to be sufficient allowance for material removal during polishing.

(iii) Bake the part for an hour or so at 100–200°C. This step is needed to improve adhesion, drive off adsorbed hydrogen and to increase the hardness. The bake temperature is chosen as high as possible to increase hardness but not so high as to compromise the amorphous character of the material[78]. That the as-deposited ELN is indeed amorphous has been confirmed by x-ray and electron diffraction and by electron and visible-light microscopy (see references in Hibbard[27]and Killpatrick[39]). If the temperature is allowed to go above about 300°C, the material transforms to a polycrystalline microstructure of Ni and Ni_3P which does not polish well.

To preserve the dimensional stability of the optical surface it is important to consider the stresses at the ELN-substrate interface. These are due to three effects (i) the intrinsic stress of the as-deposited coating (which becomes more compressive with increasing phosphorus concentration), (ii) the effect of the thermal-expansion mismatch as the part is cooled from the hot bath temperature to room temperature and (iii) the shrinkage, amounting to around 0.1-1.0 % volume change, that takes place at the bake. The latter is an irreversible one-time effect provided the coating never again sees a temperature as high as the bake temperature. Without countermeasures, the net stress can be large (100-200 MPa is not unusual) and may distort the optic or flake off the coating. To model these processes we would need to know the expansion-coefficient-versus-temperature curves of the substrate and the ELN. The latter is more complicated than it seems because both the height and slope of the ELN expansion curve vary with both phosphorus concentration and bake temperature[27]. However, by observing the curvature of plated witness strips, Parker has measured the final stress as a function of phosphorus concentration for various substrate materials[63, 64] for a bath temperature near 90°C and a bake at 140-190°C. The broad picture that emerged was that, for materials with expansion coefficients not too different than ELN; 1090 carbon steel, 304 stainless steel, beryllium, brass and nickel, low final stress could be obtained with phosphorus concentrations in the range 9-11% and therefore of good polishability. Recent experience would add Glidcop™ and certain aluminum bronzes to this list. On the other hand, for materials with high expansion coefficients such as aluminum, there are different solutions. Low stress can still be achieved but at lower phosphorus concentrations (5-7%) and bake temperatures (100-150°C) or by using a partially exhausted bath. Such coatings on aluminum can still be polished well[14] and in any case, rigorous elimination of stress is not necessary for many beam-line optics which, in the absence of weight limits, can easily be designed stiff enough to resist distortion. The opposite problem is involved in coating low-expansion materials and the stresses then tend to be tensile which is more damaging. However, by use of higher phosphorus concentrations, stable coatings can still be produced and can also be superpolished[31, 65] which is of particular importance for materials like invar and superinvar.

8.5. Glidcop™[73]

In the late 1980's, the Berkeley group made a number of Glidcop™ optics for the Advanced Light Source beam lines which met or exceeded their optical specifications and were predicted by detailed finite-element calculation to meet their thermal distortion specification. Now eight to ten years later, these optics are still performing according to expectations. The key elements involved in making cooled optics of the quality needed for this project were the thermal/mechanical engineering of the substrate[17, 18] ELN plating[1], optical working[9], ruling (in the case of gratings)[50], whole-surface metrology[79] and the integration of these into a real-world solution[49]. ELN-plated Glidcop™ is thus a major option to be considered for cooled optics and we now turn to discuss the materials issues involved.

Copper in its pure state is too weak for many applications and among the options for strengthening it are cold work and precipitation hardening. Both of these confer significant strength but in neither case does the strength survive high temperature processes such as brazes which are essential in engineering a cooled optic. The solution is dispersion strengthening which involves introducing a second phase consisting of small particles of size 5–15 nm which are stable at least up to the highest brazing temperature of about 1000°C. The alloy normally used for cooled optics (Glidcop™ AL-15, UNS C15715) has 0.3 weight % Al_2O_3 which, by limiting the movement of dislocations, allows the retention of a cold-worked microstructure and almost all of the room-temperature strength even after brazing. Recrystallization can be made to take place but only after very extended annealing at temperatures just below the melting point[77]. The material is normally purchased in the form of extruded bars which contain a skin of OFHC copper about 1.5 mm thick. This skin can be removed by machining or be exploited to simplify brazing schemes. The 0.2%-yield strength of the as-supplied material, often substantially cold-worked and highly stressed, is usually in the 300-500 MPa range while measurements have shown the stress for a strain of 10^{-4} to be 240 MPa. This measurement suggests good microyield behavior and, although no dimensional stability data on Glidcop™ are available, it fits the general description of a stable material and the experience with the ALS optics confirms, that, provided it is stress relieved by brazing or otherwise, it is stable under beam-line service conditions. Brazing of Glidcop™ can be accomplished[70] by standard methods, both to itself and to pure copper or stainless steel. A low-oxygen version of the material is recommended for brazing and we have also found that a certain amount of practice is required. A high-temperature braze can be made with (70%Ti, 15% Cu, 15% Ni) according to the following procedure; (i) 25°C/min to 900°C, hold for 15 min, (ii) 5°C/min to 980°C, hold for 5 min and (iii) cool at 25°C/min to 900°C and 5°C/min to room temperature. Substantially lower temperatures are used for silver-based brazes but a copper or nickel plated layer on the joint surface is needed to prevent rapid diffusion of the silver into the Glidcop™ bulk via its plethora of grain boundaries. Gold-copper brazes are effective without plating but a braze foil must be preplaced in the joint.

8.6. 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 due to: poor polishability of the nickel, bi-metallic bending and dimensional instability of the substrate material. Fortunately there has been considerable progress in recent years on all of these issues as we discuss below so that we can now consider aluminum[8] for a much higher quality type of optic.

The candidate materials (discounting castable alloys on UHV considerations) are now fairly well-established to be the wrought alloys 6061-T6 or T7 and the 5000 series especially 5083, and 5086 and powder-reinforced metal-matrix composites. 6061 is a magnesium-silicon alloy which is normally used in the T6 or precipitation-hardened

Table 5: Thermal cycling treatments for stabilization of aluminum alloy optics[a]

Material	**After rough machining**	**Cycle high temperature**	**Cycle low temperature**	**Total # of cycles**	**Notes**
6061	Solution treat at 530, quench in PG[c], overageage 175, 8-16 hr in stages (T7 temper)	150 for 0.5 hr	<-40 for 0.5 hr	>3	Rate <3°C/min (<1°C/min during figuring)
SXA	Solution treat at 495, quench in PG[c], age at 190 for 12 hr	180 for 0.5 hr[b]	Quench to -195[b]	until no dimension change	These cycles after finish machining
SXA	Same	170 for 4 hr[b]	-75 for 0.5 hr[b]	until no dimension change (>5)	These cycles after ELN plating, rate <5°C/min
5000 series	Anneal at 350, slow cool	150 for 1 hr	<-40 for 0.5 hr	>3	Rate <3°C/min (<1°C/min during figuring)

[a] all temperatures in °C

[b] cycling as recommended by manufacturer

[c] PG=20% polyalkylene glycol solution

condition. The 5000 series are magnesium alloys which are not heat treatable. Both types are good for welding and machining by conventional, diamond and electric-discharge machines and both have demonstrated good dimensional stability after approriate heat treatment and thermal cycling see (Table 5). However, 6061 can be brazed while, apart from 5050, the 5000 series cannot. Both have the excellent thermal conductivity and the large thermal expansion coefficient typical of most aluminum alloys. Examples of "optical grade" metal-matrix composites are SXA[2, 56] and Lanxide[40]. SXA has a matrix of 2024-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 ELN and it also gives improved microyield and creep properties. A measurement of the dimensional stability of four samples of SXA, heat treated according to the manufacturer's specification, was made by S. Jacobs[34] which showed an average shrinkage of 5.7±1.0 parts per million (ppm)/year. Generally, SXA can be made stable, but only after careful heat treatment and extensive cycling (see Table 5). It is machinable, although with difficulty, but its most serious disadvantage is the difficulty of welding and brazing. One solution is apparently to electroplate the mating surfaces with nickel and join them using tin-based solder.

It now appears that, with appropriate attention to heat treatment, ELN-plated aluminum alloys achieve dimensional stability similar to that of directly competing materials such as steel alloys and Glidcop™. Given that the polishability of the ELN should be the same and the expansion coefficient is only about 50% worse, aluminum alloys should be competitive with Glidcop™ for many cooled mirrors. Overall they should be superior to it for large mirrors, especially for cylinders needing large waster plates and for benders where the specific stiffness and total weight are issues.

8.7. 17-4 PH precipitation-hardening stainless steel

There are two reasons why 17-4 PH stainless steel deserves special consideration as an optical-substrate material. First, it has been demonstrated[16] that the *bare steel* can be polished to 2-3 Å rms roughness (measured by the optical profiler as described in section 4). Bent elliptical mirrors using these surfaces have already been used at the ALS to form a <2–micron focused x-ray microprobe. So far it appears that, apart from ELN (which can only be used as a plated layer), this is a unique capability among metals. Second, the material achieved the lowest average dimensional changes (<0.05 ppm/year) out of fifteen ceramic and metal candidate-gage-block materials[51, 53]. To these special qualities can be added an impressive array of engineering properties which are based on the principal that the material is supplied and fabricated in the solution-treated condition and then aged at a moderate temperature (480°C) allowing yield strength levels of 1.3 GPa (185 ksi) to be obtained via precipitation hardening. Other characteristics are good corrosion resistance, manufacturability similar to 300 series stainless steels, capability to be air quenched from the solution temperature even for large sections, good fatigue properties and creep resistance and a small (0.05%) and predictable contraction on hardening. Overall, the properties allow conventional UHV manufacturing methods to be applied. The alloy is designed so that the martensite transition range is just above room temperature so the air quench produces a soft low-carbon martensite which is the form used for manufacturing processes. Subsequent aging produces precipitates of copper metal that are coherent with the iron lattice and provide precipitation hardening.

The ALS microprobe mirrors, which demonstrated the 2-3 Å rms surface finish, were treated according to the following procedure:

1. Solution treat at 1050°C for 0.5 hours, air cool.
2. Cold finish to bar product.
3. Machine to size.
4. Age at 480°C for 1 hour (to condition H900), air cool.
5. Fine grind back and front surfaces.
6. Thermally cycle slowly to -196°C and 200°C, total of three cycles
7. Lap to remove about 25 micron both sides and polish.

The main question of course is why does this material polish to a superfine finish? It is hard to be certain about this but we offer the following general comments.

- The roughness of polished metal surfaces is related to differing material removal rates among the grains due either to the presence of more than one type of grain (multiphase materials) or to variations in grain orientation. In a single-phase material, such as we dealing with here, only the latter should be possible. However, unintended variability of the surface mechanical properties can still occur through the presence of inclusions. These could be minimized by purchasing "vacuum remelted" material, although that was not done in the cases reported here.
- The precipitation hardening process does not, in itself, lead to measurable roughness because the size of the particles is about 5-10 nm which is too small to see in a metallographic microscope or on the optical profiler which is essentially a visible-light interferometer. We await atomic-force micrographs and x-ray scattering measurements to understand the surface better and assess it as a multilayer substrate. However, a low roughness in the spatial frequency range of the optical profiler is enough to guarantee good performance as a soft-x-ray grazing-incidence mirror.
- In view of the considerable amount of cold work involved in "cold finishing" the bar from which these samples were taken, we suppose that a dense and uniform distribution of dislocations was formed, thus providing nucleation sites for the formation of precipitation particles. Such a dense and uniform distribution of precipitate would tend to dominate the strength properties of the grains leading to a substantially orientation-independent response to material removal during polishing. If this explanation is right then the decision not to apply a solution treatment (other than the one during manufacture of the bar) was correct and played an important role in achieving good polishability.
- The influence of grain orientation would be further weakened by the effect of the very large amount (23%) of substitutional additives which are not precipitated during hardening and which have a randomizing effect on the directionality of the elastic properties of the host lattice.

Another important question is why is 17-4 PH stainless steel dimensionally stable?[47]. A high alloy steel like 17-4 PH, with about 27% of alloying elements, has several advantages with respect to dimensional stability compared to low alloy steels of similar strength such as type 4340 which has only about 3% of alloying elements.

1. The absence of the diffusion-controlled transformations to pearlite and bainite in a martensitic precipitation-hardening stainless steel, allows the use of very slow cooling rates during heat treatment. This reduces residual stresses and warpage due to the martensite quench.
2. The age-hardening temperature to produce condition H900 (480°C) is sufficient to produce a significant degree of stress relief.
3. The low carbon reduces the chance of retained austenite after quenching and diminishes the importance of carbide precipitation and carbon migration processes (see section 3.2).
4. The martensite, which is supersaturated with *substitutional* alloying elements, has a specific volume which differs less from the stable bcc structure than it would do if it was supersaturated with carbon which is an *interstitial* alloying element. Thus the volume change due to precipitation reactions which happen either rapidly during aging or gradually over time is much less.

We believe that the ability to superpolish stainless steel has the potential for revolutionizing the way many synchrotron radiation mirrors are made. This will not apply to intensively-cooled mirrors, which need to be made from good thermal conductors such as Glidcop™ or silicon, but it could apply to many mirrors which are not intensively cooled. For moderate cooling, standard UHV manufacturing techniques can be implemented by any competent machine shop at reasonable cost and low technical risk. The use of metals for making mirror substrates has always been beneficial on cost and engineering grounds. With this material their two main disadvantages; dimensional-stability concerns and nickel-plating problems, are eliminated.

8.8. Mild steel 1010

Another approach to dimensional stability, used with some success in the gage-block experiments, is to use a fully-annealed plain-carbon steel with very low carbon. This gives optimum stress relief, eliminates all of the martensite-tempering-type reactions and provides a simple low-cost substrate with a good thermal match to ELN. Since this material is practically pure iron, it has a much better thermal conductivity than high alloy steels. In addition, (like 17-4 PH) it is magnetic and thus easy to surface grind. The microyield stress will be somewhat reduced by the anneal but moderate bending stresses can still be tolerated. A large ($1.25 \times 0.10 \times 0.015$ m^3) ELN-plated mirror has been made for the ALS[9] by this approach and was formed into a 10:1 demagnifying elliptical cylinder by bending with a maximum stress of 20 MPa. The intended shape was reproduced within 3 μr rms over the center 0.6 m and within 15 μr rms over 1m. Most of the errors were traced to an initial curvature of about 0.5 km that could not be removed by either lapping or polishing. Such a curvature produces errors in mirrors which are shaped to bend into noncircular shapes. We conclude that, although the errors were gratifyingly small for such a difficult mirror, it appears that improvements are still possible by attention to the initial preparation of the blank.

8.9. Invar and superinvar

Invar and superinvar have the best thermal-distortion performance in the temperature range 0-80°C of all the materials considered here plus wide availability and many of the advantages of a steel alloy. Such advantages include the capability to be welded, brazed and machined without major deviations from the normal practices for dealing with steels. These are very significant advantages but they can only be exploited if the major issue of dimensional stability can be addressed. However, great deal is known about the length-change behavior of invars for both varying and constant temperature[33, 48, 69] as we discuss below.

The low expansion properties of the nickel-iron alloys were discovered by Charles Guillaume in 1886 [25, 26]. 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. 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 suffers from temporal instability. 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 Cohen[43]. 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%, then its presence as graphite must be avoided or it will raise the coefficient of thermal expansion. This can be accomplished by a solution treatment at 830°C followed by a quench. 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 "triple treatment") that has received wide acceptance in the literature as providing the best combination of low α and good dimensional stability:

(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.

There are also some useful variations to the triple treatment to be considered. First, if stress-inducing treatments such as machining and grinding are required then step (ii) can be done in two parts, one after rough machining and a second after finish machining. Some thermal cycling (below 315°C) can also be inserted after both finish machining and lapping. Once the final aging step (iii) has been done, that temperature must never be exceeded. Second, it has been shown that both dimensional stability and microyield stress are improved when polyalkylene glycol is used as a quenchant instead of water.

The data on just how good the expansion coefficient and length stability can be after the triple treatment are sparse and somewhat contradictory. Steel et al.[76] have 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. The fact that there appears to be both a fast and a slow growth rate allows at least the possibility to reconcile the apparently conflicting literature. 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 0.15–1.0 ppm/°C for temperatures 0–80°C[83] and stabilities of 2–4 ppm/year. Moreover, if manganese is low, an expansion coefficient of 0.15–0.4 ppm/°C and if carbon is low, a stability of 1–2 ppm/year may reasonably be expected.

There are two ways to improve on commercial invar. One is to seek an invar alloy with very low carbon and manganese. Such an alloy would be free of both the γ expansion and graphite precipitation and this has been the thrust of recent developments by the Jet Propulsion Laboratory (JPL)[75] based on the use of powder metallurgy to achieve a highly controlled composition with carbon below 0.01%. The triple treatment was applied but with a slow cool from the solution temperature. The invars produced in this way have been tested by Jacobs and have shown expansion coefficients below 1 ppm/°C and stability better than 1 ppm/year[75]. A second way is to use superinvar. This material can provide an expansion coefficient of 0.05 ppm/°C over 0–80°C and a length stability $\leq$2 ppm/year using heat treatments the same as for regular invar. Its principal difference from normal invar is that the replacement of about 5% of the nickel with cobalt raises the martensite start temperature to a nominal value of –80°C. This value is extremely sensitive to the carbon and silicon impurity content and it is wise to avoid aggressive cold treatments of this material unless the carbon and silicon are extremely low.

For comparison with the values discussed above, "standard" fused silica has α=0.6 ppm/°C and a length change rate of 0.2 ppm/year. Thus the expansion and stability figures of invar can certainly be brought within the range needed for high quality optics. Of course the question of polishing still remains and there are two approaches: polishing of the bare invar and polishing an ELN coating. We have carried out tests on polishing bare invar[32]. with quite encouraging results. The surface roughness values, defined as described in section 4, were in the range 7–10 Å rms for seven 50-mm-diameter test pieces. The successful achievement of a superpolish on ELN-plated invar has been explained in section 8.4. However, we should note that the ELN bake should precede the final aging treatment of the invar.

9. 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 is to use nickel-plated glidcop or single-crystal 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 have discussed how we may 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 with efforts to include pin-post and cellular-pin-post systems. When the emphasis is on complying with extremely tight distortion specifications then it appears that low-expansion materiuals such as silicon are indicated and that invar offers a possibility 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 certain aluminum and stainless steel alloys can be added to Glidcop™ and invar as candidate materials. In our opinion, once internal-cooling designs using these materials are established, they will be seen as equally cost-effective and more 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, silicon and the glasses have the advantage. From this analysis it appears that, although silicon carbide offers certain capabilities, it is essentially never the simplest and most cost effective solution to a beam-line mirror. 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.
- Develop a way to finish ELN that is compatible with multilayers, i. e. achieve low roughness at all spatial periods down to near-atomic dimensions.
- Pursue the polishing of bare metals especially invar and molybdenum.
- Investigate polishable coatings that can be applied to desirable substrate materials, for example, sputtered molybdenum on molybdenum or physical-vapor-deposited silicon on invar.

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