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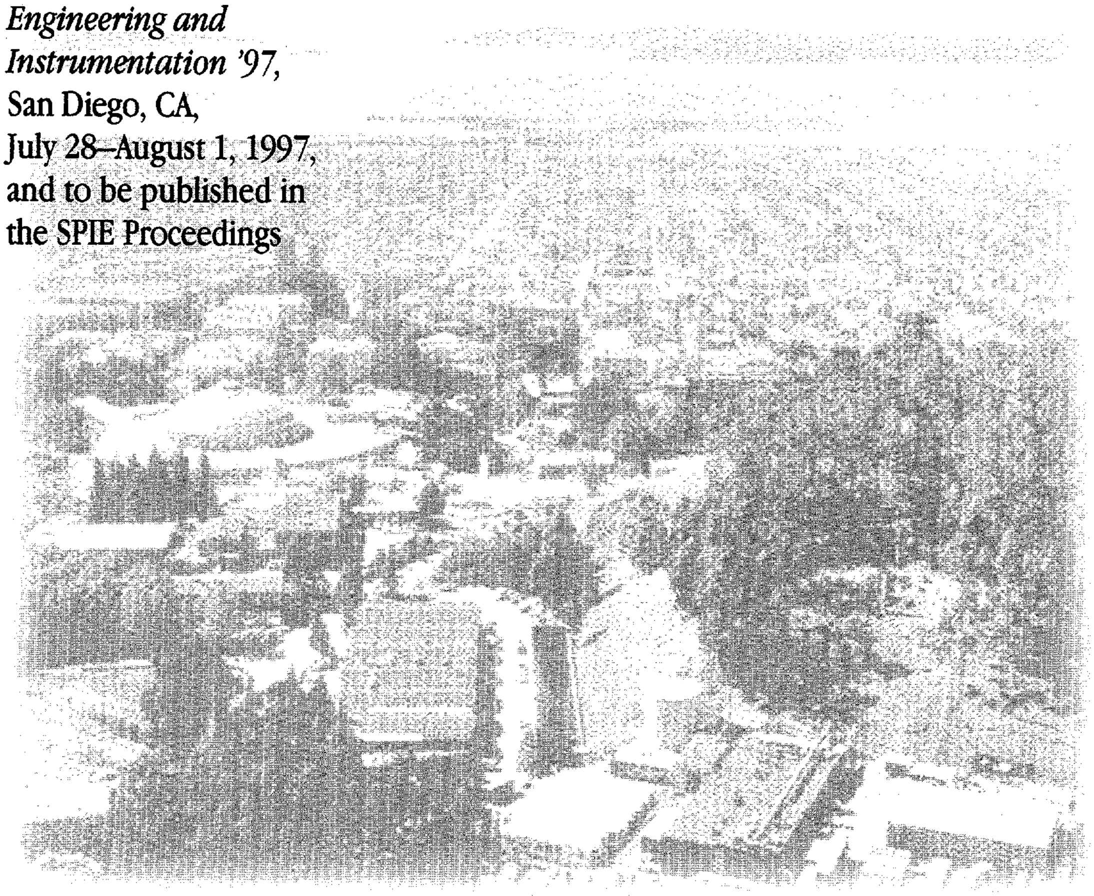

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ACHIEVEMENT OF A SUPERPOLISH ON BARE STAINLESS STEEL

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Achievement of a superpolish on bare stainless steel

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ABSTRACT

We report the achievement of a superpolished surface, suitable for x-ray reflection, on bare stainless steel. The rms roughness obtained on various samples varied from 2.2 to 4.2 Å, as measured by an optical profiler with a bandwidth 0.29-100 mm^{-1}. The type 17-4 PH precipitation-hardening stainless steel used to make the mirrors is also capable of ultrastability and has good manufactureability. This combination of properties makes it an excellent candidate material for mirror substrates. We describe the successful utilization of this type of steel in making elliptical-cylinder mirrors for a soft-x-ray microprobe system at the Advanced Light Source, and discuss possible reasons for its unusual stability and polishability.

Keywords: Superpolish, x-ray mirror, metal mirror, precipitation-hardening stainless steel, synchrotron radiation.

1. INTRODUCTION

Mirrors for synchrotron-radiation beamlines normally operate in ultrahigh vacuum (UHV) and are illuminated by x-ray beams which may deposit significant power into the mirror substrate. These grazing-incidence reflectors are critical components in the application of synchrotron radiation, and considerable effort has been devoted to their design and manufacture[1-4]. Generally there are numerous thermal, mechanical and optical requirements to be satisfied, and design approaches using both metallic and ceramic substrates have been developed. The balance of issues involved in choosing a substrate material is complex[3], but the broad picture is that appropriate ceramic substrates are easier to polish but more difficult to engineer, while suitable metallic substrates are easier to engineer and more difficult to polish. In fact, until now, the only metallic material that could be polished to the superfine finish required for x-ray work has been electroless nickel, so that all metal mirrors for synchrotron-radiation beamlines have necessarily been fabricated with electroless-nickel coatings, usually of around 100 microns thickness. Such coatings pose significant costs and risks, and the necessity for using them on metal substrates has been one of the principal reasons why the community has tended to prefer ceramic mirror substrates over metal ones.

In this paper we report significant progress in producing superpolished metal surfaces *without* the use of electroless nickel coatings. The material we have used is 17-4 PH precipitation-hardening stainless steel (also known as Custom 630™), which has excellent ultrahigh vacuum and general engineering properties, as well as an unusual degree of dimensional stability. In what follows we present evidence demonstrating that a superfine finish has been consistently achieved on this material both in our polishing tests and in several x-ray microprobe mirrors made for the Advanced Light Source (ALS)[10]. We also provide further description of the properties and microstructure of the alloy and a discussion of possible reasons why it has such exceptional dimensional stability and polishability.

2. MOTIVATION

The use of high-strength materials such as 17-4 PH stainless steel for mirror substrates is especially interesting for mirrors that will be formed by bending to a high curvature. In such cases, ceramic materials like glass can only be used if the stress is kept low by using a very *thin* substrate. This leads to a mirror that is not sufficiently rigid for high-quality polishing. On the other hand, high-strength steel alloys can tolerate much higher stresses without breaking or yielding, thus allowing mirrors to be thicker and therefore more polishable. In addition, the problem of joining a mirror to its bending machine is very simply solved in the case of steel mirrors by using nuts and bolts. The argument so far does not rule out an electroless-nickel-coated steel mirror. However, in the microprobe application to be described later, one of the mirrors had to be bent to 4.5 m radius, corresponding, in our case, to a bending stress of 150 MPa. Moreover, we know that electroless-nickel coatings are likely to have significant intrinsic stresses resulting from deposition conditions[3] which could add to the applied bending stress. Given this background, we did not believe that electroless-nickel coatings would perform reliably for this type of application, and this was the primary motivation for experimenting with uncoated metal mirrors.

3. GENERAL ALLOY PROPERTIES

The percentage constituents of 17-4 PH stainless steel are (C 0.07, Mn 1.0 max, P 0.04 max, S0.03 max, Si 1.0 max, Cr 15-17.5, Ni 3.0-5.0, Cu 3.0-5.0, Nb+Ta 0.15-0.45, Fe balance). Its advantageous properties are obtained[11] in part by designing the alloy with a martensite transition range just above room temperature. In the absence of the transitions to pearlite or bainite, the alloy may be cooled slowly from the solution-treatment temperature even for thick sections. The result is that a soft low-carbon martensite is formed which is relatively easy to machine and fabricate. Hardening to "condition H900" with a tensile strength of about 1.4 GPa (44 Rockwell "C") is then accomplished by a mild aging treatment at 480°C for 1 hour, which precipitates extremely small (<10 nm) crystals of copper metal which are coherent with the host lattice. The aging process does lead to dimensional changes, but they are small (about 0.05%) and predictable. The high dimensional stability of 17-4 PH stainless steel, together with its high strength and creep resistance, promises excellent performance as a mirror substrate. Moreover, the corrosion resistance and fabrication characteristics, prior to aging, are similar to those of commonly-used stainless steels such as type 304, so that compatibility with the beam-line environment is also assured.

17-4 PH is a member of a family of precipitation-hardening stainless steels, and we believe that other members of the family such as 15-5 PH may work equally well as optical substrate materials. However, there is also a separate family of hardenable stainless steels of comparable strength to 17-4 PH, but with much higher carbon. Such alloys are generally hardened by *quenching* from the solution temperature. As we discuss later, we believe that both the high carbon and the need for a quench are very unwelcome in an optical component, and that alloys that require them are much less desirable.

4. POLISHING AND TESTING

Seven test samples in the form of 50-mm diameter disks 12 mm thick and nine mirrors for the microprobe project (120×40×6 and 75×25×3 mm^3) were manufactured at Lawrence Berkeley National Laboratory (LBNL) according to the following procedure:

1. Solution treat at 1050°C for 0.5 hours, air cool.
2. Form and hot or cold finish to bar stock.
3. Machine to size.
4. Age at 480°C for 1 hour, 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, removing at least 20-30 microns, and polish.

The samples were then sent to Dallas Optical Systems for polishing and were subsequently evaluated using the Micromap Promap 512 optical profiler at LBNL. This instrument has a 2.5× objective and a bandwidth of 0.29-100 mm^{-1}. The Micromap trace of a typical example of the set is shown in Fig. 1. The general appearance of the measured data for all seven samples was very similar to the one shown, and the seven values of the roughness rms height were 2.84, 2.63, 2.64, 2.48, 2.62, 2.68, and 2.58 Å. The general shape of the curves and the values of the parameters which characterize them are broadly similar to those that we have measured for other optics made for the Advanced Light Source that have met their specifications and been successful in service. Subsequently, the microprobe mirrors were tested similarly with basically identical results. The spread of values of the rms roughness was a little higher, 2.2-4.2 Å, but most were concentrated within the same range.

We have also initiated a series of measurements of the high-spatial-frequency roughness of the polished surfaces using soft x-ray scattering and atomic-force microscopy. Preliminary indications are that the present level of high-frequency roughness is too high to allow the use of these mirrors as x-ray multilayer substrates. This is in agreement with expectation, since one would expect to see surface roughness on the spatial scale of the copper precipitate particles (≈10 nm) which would not affect performance as a specular reflector, but would perturb the interfaces of a multilayer to an intolerable degree.

5. METALLOGRAPHIC MEASUREMENTS

The martensitic structure of the 17-4 PH alloy is formed just above room temperature. Therefore one expects to see the low temperature form which is plate or lenticular martensite. A micrograph of the (etched) surface of one of the LBNL mirror samples is shown in Fig. 2. The appearance is very similar to handbook micrographs of the same material[5] with identical heat treatment and etching (in Fry's reagent). It appears that the grains are quite large, 40-100 µm, while the finer structures visible in the micrograph, at the few µm level or larger, are due to twinning within the grain. We examined both a cold-finished 50-mm-diameter bar from which the disk samples were cut and a hot-finished 200-mm-diameter bar from which the microprobe mirrors were cut. All the polished mirrors were cut transversely. We looked at both the polished mirrors and metallographic samples that had been cut both longitudinally and transversely to the bar axis and from both the center and outer regions of the larger bar. No differences were seen on account of these different cuts. This encourages us to think that we may be able to make long narrow mirrors by cutting them *lengthwise* from the bar stock.

6. APPLICATION OF THE STEEL MIRRORS TO AN X-RAY MICROPROBE BEAMLINE AT THE ALS

The microprobe was to be formed by focusing the x-ray beam with a pair of mirrors at 1.6° grazing angle in Kirkpatrick-Baez geometry. Two types of flat mirror were constructed as described in section 4. Each had a thickness that was uniform within 1-2 μm and a width that varied according to a calculated function that was designed to produce a desired elliptical shape upon bending by end couples of the proper value[9,10]. The delivery of accurately controlled bending couples to the mirror ends was accomplished by a method developed by the Berkeley group using weak leaf springs[9,10] (Fig. 3). The front of the mirror was polished by Dallas Optical Systems while the back was lapped to a sufficient flatness that it remained within tolerance when it was bolted to the (similarly lapped) mating surfaces of the bending springs. After due attention to assembling the bender and springs without unacceptable twisting of the mirror, the procedure described by Rah[12] was followed to choose the values of the couples for best fidelity to the desired ellipse. The final mirror shapes followed their intended ellipses within 2-3 μr, which enabled x-ray spot widths of 1.5-2.0 μm to be obtained at 1 keV in micro-XPS experiments. The majority of the mirror errors contributing to the spot width came from lack of flatness before bending.

7. DISCUSSION OF POLISHABILITY

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 normally 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 are dealing with here, only the latter should be possible, although unintended variability of the surface mechanical properties could still occur through the presence of inclusions. Such inclusions may be minimized by purchasing "vacuum remelted" material, although that was not done in the tests reported here.
- The precipitation hardening process does not lead to roughness in the frequency range that is measured by the optical profiler (0.29-100 mm^{-1}) and this is (intentionally) about the same as the range involved in grazing-incidence specular reflection of x-rays. So the Micromap readings alone are enough to guarantee good low-scatter performance as a soft-x-ray mirror. As indicated earlier, the precipitate particles in the 5-10 nm range do give higher frequency roughness which, for the present, will probably prevent these mirrors from being used as multilayer substrates.
- In view of the considerable amount of area reduction involved in cold or hot finishing of the bar from which these samples were taken, we suppose that a dense and uniform distribution of dislocations was formed, which would be expected to nucleate a corresponding distribution of precipitate particles during hardening. 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 our 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.

8. DISCUSSION OF DIMENSIONAL STABILITY[6]

The most systematic investigation of dimensional stability was the study of potential gage-block materials by the U.S. National Bureau of Standards in the 1960's. 17-4 PH stainless steel achieved the lowest average dimensional change (<0.05 ppm/year) out of fifteen ceramic and metal candidates that were tested[7,8]. 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.

- The absence of the diffusion-controlled transformations to pearlite and bainite in a martensitic precipitation-hardening stainless steel allows a slow cooling rate (i. e. not a water quench) from the solution temperature. This reduces residual stresses and warpage due to the martensite transformation.

- The age-hardening temperature to produce condition H900 (480°C) is sufficient to produce a substantial degree of stress relief.
- The low carbon, coupled with the effect of the thermal cycling, reduces the chance of retained austenite after the martensite transformation and diminishes the importance of carbide precipitation and carbon migration processes.
- 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. The larger lattice distortion associated with its interstitial position appears to be the main reason why carbon plays such an important role in dimensional stability and why its reduction or elimination is so beneficial for such stability. The lattice parameters of austenite, martensite and ferrite all depend linearly on the carbon concentration, as do the specific volumes of carbon-containing microstructures such as pearlite and bainite. Moreover, the dependence is on the order of one to one, meaning that the specific volume changes by about one per cent for each one per cent change in carbon concentration. When dimensional changes of fractions of a part per million are considered significant, carbon movements will obviously be very important. According to this, martensite-tempering-type reactions[13] involving carbon will tend to dominate the dimensional-stability behavior of steels, all of which confirms the importance on minimizing the carbon.

9. DISCUSSION OF FUTURE PROSPECTS FOR NEW WAYS TO MAKE SYNCHROTRON RADIATION MIRRORS.

We believe that the results reported here have 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 the majority of mirrors are not in this category. Now that steel mirrors can be polished without the use of electroless-nickel coatings, the cost and risks associated with these coatings can be avoided. In the case that moderate cooling is needed, 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. Now it appears that the two main disadvantages of the approach, dimensional-stability concerns and nickel-plating problems, can be eliminated in a large number of cases.

10. ACKNOWLEDGEMENTS

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12. FIGURES

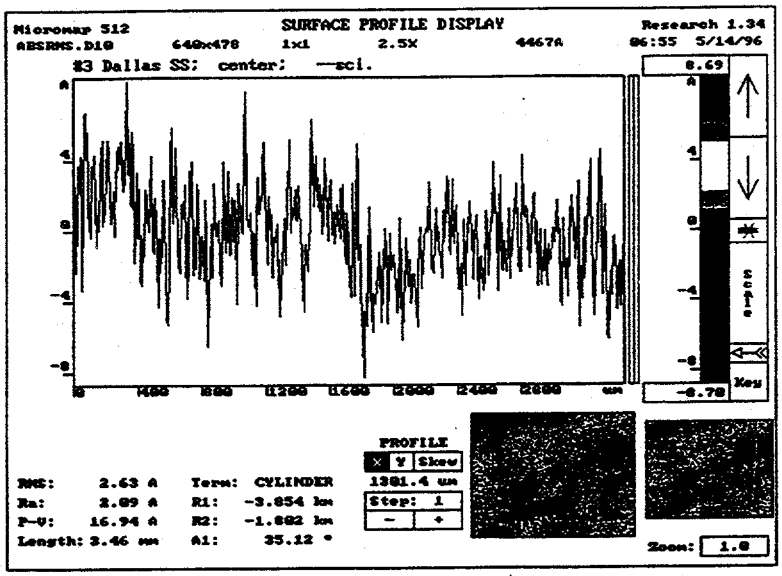

Fig. 1. Micromap trace of a typical member of the initial set of seven test pieces

Fig. 2. Optical micrograph of the surface of an aged sample of 17-4 PH stainless steel etched in Fry's reagent (magnification=400×)

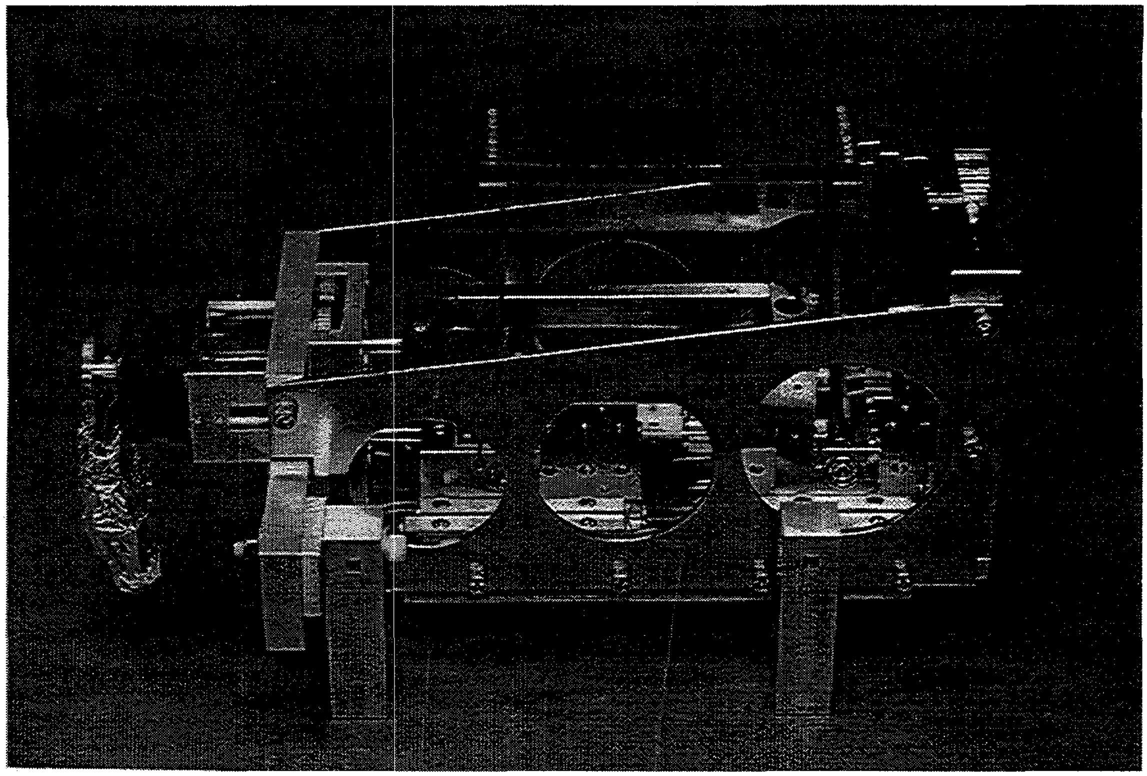

Fig. 3. View of an installed steel mirror bolted to the springs which deliver the bending forces producing the elliptical shape. The center bolts are inserted from below to allow the grazing-incidence beam to pass. The clear aperture of the mirror begins a few thicknesses in from the row of bolts to allow the distortions due the bolt forces to diminish.