MIRROR SUBSTRATE MATERIALS FOR SYNCHROTRON RADIATION BEAM LINES

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ADVANCED LIGHT SOURCE

MIRROR DESIGN OVERVIEW

- Tasks vary according to specific requirements and tolerances but there are general issues that always arise

- 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? Should the mirror be rigid or bendable

- Material quality: can the material be obtained in the required quality and size and will it hold its shape over long times?

- Cost: single beam lines (not including the undulator if there is one and the experiment) cost $1-5M. Are the optics taking a "reasonable" share?
  (They are the only components the *x-rays* ever see!)

POLISHABILITY OF MATERIALS

- Materials that can be finished (superpolished) to a low scatter surface:

  Defined as Š5 Å rms roughness measured with an optical profiler with a bandwidth of at least 0.3 - 100 mm^{-1}

  Glasses:
  - Fused silica
  - ULE™ fused silica
  - Zerodur

  Crystalline ceramics:
  - Intrinsic single-crystal silicon
  - CVD beta silicon carbide

  Metals:
  - Any metal coated with electroless nickel (EN)!
  - 17-4 PH stainless steel

- Materials that can be finished well enough to take an x-ray multilayer coating:

  - Silicon (poly and single crystal)
  - Glasses

- Materials that can be finished <u>almost</u> to a superpolish:

  - Invar
  - Molybdenum
  - Reaction-bonded silicon carbide

OPTICAL SPECIFICATIONS

- Ideally we should specify a bound on the power spectral density function for all frequencies from atomic scale to mirror-size scale - this is possible to do from theory but opticians cannot make power spectra to order - therefore we compromise in practice as follows

- Figure specification:
- Tangential rms slope error equals one quarter of the angular subtense of the source (or slit) - one factor of a half because $\sqrt{1^2 + \left(\frac{1}{2}\right)^2} \cong 1.1$ and one because the ray rotation is twice the mirror rotation
- Sagittal rms slope error equals the same thing divided by sine of the grazing angle (forgiveness factor)
- Compliance to be verified by interferometry or the long-trace profiler (Takacs 1988)
- In the range 0.3-3 µr rms for first quality optics

- Finish specification:
- Š6Å rms for first quality optics
- Optical profiler readings of around 1 Å rms or less are often achieved and are a necessary (although not a sufficient) condition for good multilayer operation
- Compliance to be verified by an optical profiler with a bandwidth of 0.3-100 mm^{-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	

OPTICS, CONSEQUENCES AND MATERIALS

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	Polishability: ceramic preferred over metal but ELN and
	Finish spec. roughness	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 figure of merit
	Thermal cycling spec.	Fatigue endurance limit
Toleration of gravity	Gravity distortion spec.	Specific stiffness
UHV compatibility	100% dense	Low vapor pressure: substrates and adhesives
	Bakeable to 100°C	Some processes must be at higher temperature
	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
		See properties under "active bending"
	Flat-polishing-plus-bending preferred to aspheric figuring	

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

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				
Electrolyticnickel		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	

DIMENSIONAL INSTABILITIES

Type of instability	**Spontaneous**	**Thermomechanical cycle**	**Thermomechanically driven**
What the environment does	nothing	changes and returns to its initial state	changes
Examples: general	gamma expansion in invar, carbide precipitation in carbon steels	stress relief due to heat treatments or removal of stressed surface layers, microyields after load-unload cycles	"bimetallic-strip" bending, any failure to make dimensions independent of environment variables
Examples: synchrotron radiation		stress relief due to bakeout	change of surface shape of a cooled optic due to decay of beam current, creep due to intentional mirror bending

COUNTERMEASURES

- Use materials with high microyield stress capability 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.
- Avoid deposited layers such as electroless nickel or CVD SiC or design them for low stress.
- 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).
- Thermally cycle the parts a few times over a wider temperature range than they will see in service before final polishing to an optica figure. (See section on aluminum alloys.)
- If the part is to be used under stress then stress cycle it similarly.
- If aggressive heat treatments are not practical, then consider mechanical vibration or mechanical vibration plus mild heat treatment.
- If stressed layers are known to be present at the surface, for example due to surface grinding, remove them using a low-stress process such as lapping or chemical etching.
- 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.
- Example of a distortion problem caused by bakeout; ALS Glidcop™ refocusing mirror. The distortion resulted from stress relief because the mirror, being uncooled, had never been brazed which would normally have provided sufficient stress relief.

MICROYIELD AND MICROCREEP

- Microyield stress is important for precision instrumentation - it is normally quoted as the stress cycle for a plastic strain of 1 ppm
- The microcreep is the strain in a given time at a given *constant* stress - this would be the relevant quantity for a mirror bender where the stress was high (generally it is low)
- Data are not easy to find on either - right are some microyield data from the principal reference of the field
- Microyielding or microcreep involves the movement of dislocations. Thus materials in which dislocations are pinned by dispersion strengthening (Glidcop) or precipitation hardening (17-4PH stainless steel, 6061 Al) have the best properties
- Generally materials of the same class and heat treatment have microyield values that are roughly the same fraction of the 0.2% yield - for heat-treatable Al alloys the figure is about 0.6 while for non-heat-teratable or fully annealed alloys it is lower 0.3-0.4 - for Be alloys it is only about 0.15
- Generally small-grain, highly alloyed or multiphase materials have the highest ratios

Microyield stress of some materials [Marschall and Maringer 1977]

Material	Strain	MN/cm^2	ksi	Heat treatment	Comment
1015 carbon steel	1×10^{-6}	35	5	anneal furnace cool	0.15% Ti – 3.5-7% prestrain
1020 carbon steel	2×10^{-6}	660	96	harden and temper at 205°C	worse with no temper or higher temper temperature
invar	1×10^{-6}	41	6	anneal water quench	
Free-machining invar	0.5×10^{-6}	180	26		
4340	1×10^{-6}	270	39	anneal air cool	
Ni-Span-C	1×10^{-6}	260	38	Anneal, water quench, age 21 h at 675°C	Fe-42Ni-5Cr-2.5Ti-0.6Al
440C stainless	1×10^{-6}	470	68	Anneal, oil quench, temper 1 hr 260°C	
310 stainless steel	0.5×10^{-6}	156	22.7	not heat treatable	
300 maraging steel	2×10^{-6}	1840	267	hardened	
400 maraging steel	2×10^{-6}	2300	330	hardened	
6061 Al	1×10^{-6}	210	30		
356-T6 Al	0.5×10^{-6}	56	8		
5456 Al	1×10^{-6}	146	21	not heat treatable	
2024 Al	1×10^{-6}	251	36		
AZ 92A Mg	0.5×10^{-6}	35	5		
Cu-2Be	2×10^{-6}	100	15	Annealed	
Cu-2Be	2×10^{-6}	290	41	24 h at 425°C	holds to 200°C
TZM-Mo	1×10^{-6}	378	54		

TABLE 5. Summary of Stability Characteristics of All Gage Blocks, In Order of Merit

Type[a]	Symbol	Surface hardness,[b] Rc	Material and identifying treatment	Max period observed, months	Fabrication of case on nongaging faces: Intact	Partially removed	Completely removed	Not applicable	Average change in length, μ-in./in./yr
3	P	>70	17-4 PH, hardened, aged, nitrided	22			X		+ 0.05
4[c]	KA	72	Titanium carbide, 25% Ni, stress relieved	20				X	− 0.07
2	F	68	410, annealed core, nitrided	45		X			+ 0.10
4	S	65	Titanium carbide, steel binder, hardened and tempered	19				X	− 0.10
2	L	66	1010, pack carburized, case hardened	30		X			+ 0.11
1[d,e]	T	62	52100, directly quenched or martempered, stabilized, Rc 62	49				X	− 0.13
3	N	68	Nitralloy 135 mod, hardened, tempered, nitrided	34		X			+ 0.17
2	L	65	1010, carbonitrided, case hardened	20			X		+ 0.17
3	N	68	Nitralloy 135 mod, hardened, tempered, nitrided	34			X		+ 0.19
1[d,e]	T	60	52100, directly quenched or martempered, stabilized, Rc 60	50				X	+ 0.20
2	F	67	410, annealed core, nitrided 2-step	45	X				+ 0.20
3	N	57–69	Nitralloy 135 mod, hardened, tempered, liquid nitrided	17			X		+ 0.21
2[f]	T	68	52100, annealed, chromium plated	48		X			+ 0.21
2	L	62	1010, annealed core, thermal sprayed	17			X		+ 0.25
1[d]	V	62	T15, hardened, tempered to Rc 62	19				X	+ 0.27
3	E	66	8620, carbonitrided, case hardened	36			X		+ 0.29
1[d]	T	65	52100, directly quenched or martempered, stabilized, Rc 65	49				X	− 0.30
4	AL & NBS	>68	Chromium carbide, Ni binder, as received	20				X	− 0.34
2[c]	L	68	1010, annealed core, chromium plated	25		X			+ 0.35
4	Q	>70	Aluminum oxide, no binder	29				X	+ 0.38
3	E	65	8620, liquid carburized, case hardened	22			X		+ 0.38
2	N	68	Nitralloy 135 mod, annealed core, nitrided	17			X		+ 0.40
2[f]	T	72	52100, annealed, flame plated	30			X		+ 0.41
3	E	64	8620, gas carburized, case hardened	20			X		+ 0.42
2	F	68	410, annealed core, nitrided	30			X		+ 0.44
1[d]	V	65	T15, hardened, tempered to Rc 65	20				X	+ 0.49
2[c]	L	68	1010, annealed core, chromium plated	36			X		+ 0.58
1[d]	W	65	W4, hardened, stabilized	16				X	+ 0.59
1[d]	...	65	Commercial through-hardened steel gage blocks, AA grade	25				X	− 0.67
4	KB	68	Titanium carbide, 40% Ni, stress relieved	19				X	− 0.78
3[e]	C	65[g]	420, hardened, tempered, nitrided	16			X		+ 0.80
2	D	65	D2, annealed core, nitrided	16		X			+ 0.81
2	F	68	410, annealed, chromium plated	24			X		+ 0.85
1[d]	D	58	D2, hardened, stabilized	16				X	+ 0.86
2	D	72	D2, annealed, flame plated	24			X		+ 0.89
2	F	72	410, annealed, flame plated	23			X		+ 0.96
1[d]	T	66	52100, hardened, deliberately unstable	7				X	−25.0

a Type 1 is through-hardened; Type 2 has annealed cores and hardened surfaces; Type 3 has partially hardened cores and hardened cases; Type 4 is a ceramic or cermet.
b This value represents hardness of the case or coating where applicable. Where appropriate, hardness was measured with a micro-tester such as the Vickers and converted to Rc.
c Only one specimen of this type tested.
d Results reported were obtained previously and reported in a prior reference (2). They are included for comparison.
e Contained short-term periods of greater instability.
f Average of two blocks with widely different values. One block was very stable.
g Estimated.

17-4 PH STAINLESS STEEL AS AN OPTICAL SUBSTRATE: OVERVIEW

- **METALS ARE DESIRABLE SUBSTRATE MATERIALS FOR COST AND ENGINEERING REASONS BUT THEY HAVE TWO DOWN SIDES**

  - **The need for an electroless nickel layer to achieve a superpolish**

  - **Metals are not automatically dimensionally stable, need attention to this issue**

- **17-4 PH STAINLESS STEEL HAS HIGH DIMENSIONAL STABILITY (REF GAGE-BLOCK EXPERIMENTS SHOWING INSTABILITY < 0.05 PPM/YR)**

- **WE NOW KNOW THAT 17-4 PH STAINLESS STEEL CAN BE POLISHED TO 2-3 Å RMS (BY WYCO-TYPE MEASUREMENT) <u>WITHOUT</u> AN ELECTROLESS NICKEL LAYER**

- **17-4 PH STAINLESS STEEL HAS GOOD MANUFACTURING QUALITIES SIMILAR TO THE POPULAR TYPE 304**

GENERAL ENGINEERING PROPERTIES

- The material is supplied and fabricated in the solution-treated condition and then aged at a moderate temperature allowing high strength levels to be obtained via precipitation hardening.
- Good corrosion resistance
- Formable, machinable and weldable roughly similarly to 300 series stainless steels
- Air quenchable from the solution temperature even for large sections
- Good fatigue properties and creep resistance
- Mild aging treatment provides stress relief as well as strengths up to 200,000 psi (43-45 Rockwell "C")
- Contraction on hardening is small (about 0.05%) and predictable
- Overall properties allow conventional UHV manufacturing methods to be applied

17-4 PH STAINLESS STEEL: GENERAL METALLURGY

- Composition is (C 0.07, Mn 1, Si 1, Cr 15-17.5, Ni 3-5, Cu 3-4, Fe balance) chosen so that:

- The diffusion-limited transitions to pearlite and bainite do not take place so only an air cool from the solution temperature is required (no quench)

- M_s and M_f temperatures are 138°C and 38°C so the martensite transition is complete at room temperature and the solution-treated form is a soft low-carbon martensite - (retained austenite can be reduced by cold treatments)

- High strength is achieved by a mild aging treatment (480°C) which forms precipitates of Cu metal that are coherent with the iron lattice and provide precipitation hardening.

- Manufacturing procedure

1. Solution treat at 1050 °C for 0.5 hours, air cool.

2. Cold finish to 2-inch diameter bar.

3. Machine to size.

4. Age at 480°C for 1 hour (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 both sides removing -25 µm and polish.

WHY DOES 17-4 PH STAINLESS STEEL POLISH TO A SUPERFINE FINISH?

- The roughness of polished metal surfaces is related to differing material removal rates among the grains. In a single-phase material (as here) this comes from variations in grain orientation.

- The precipitation hardening process does not, in itself, pose a surface roughness problem because the size of the particles are in the 5-10 nm range which is too small to see on the finish-measuring instrument (Micromap).which is essentially a visible-light interferometer. However, the Micromap readings alone are enough to guarantee good low-scatter performance as a soft-x-ray mirror.

- Other measurements show a roughness on the scale of the precipitate particles which will prevent this material being used as a multilayer substrate.

- In view of the considerable amount of cold work involved in "cold finishing" the bar, we expect a dense and uniform distribution of dislocations providing a corresponding distribution of nucleation sites for the formation of precipitation particles during hardening. Such a 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.

WHY IS 17-4 PH STAINLESS STEEL DIMENSIONALLY STABLE?

1. The absence of the diffusion-controlled transformations to pearlite and bainite in a martensitic precipitation-hardening stainless steel, in other words its high hardenability, allows the use of very slow cooling rates during heat treatment. This reduces residual stresses and warpage due to the martensite quench.

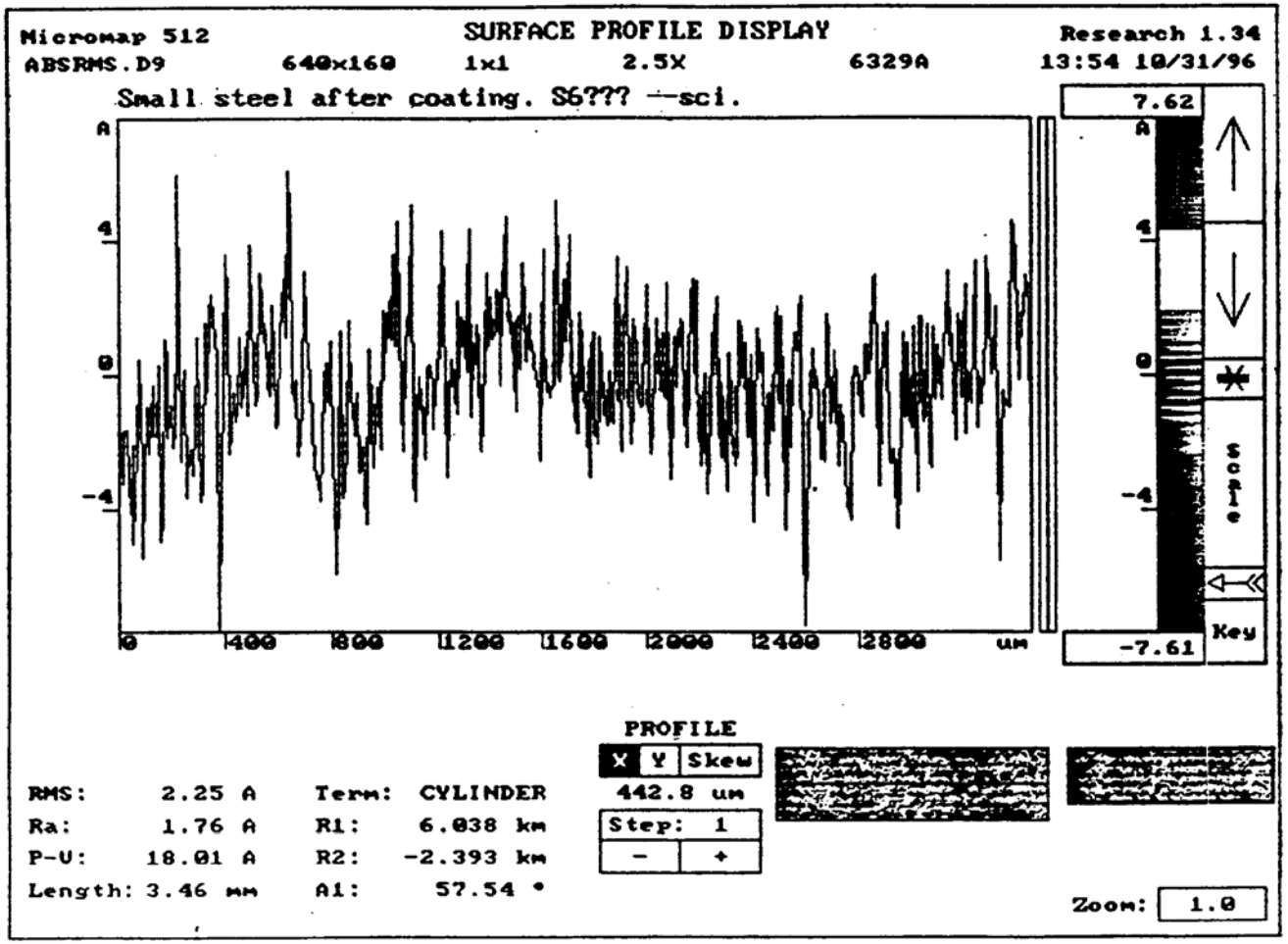
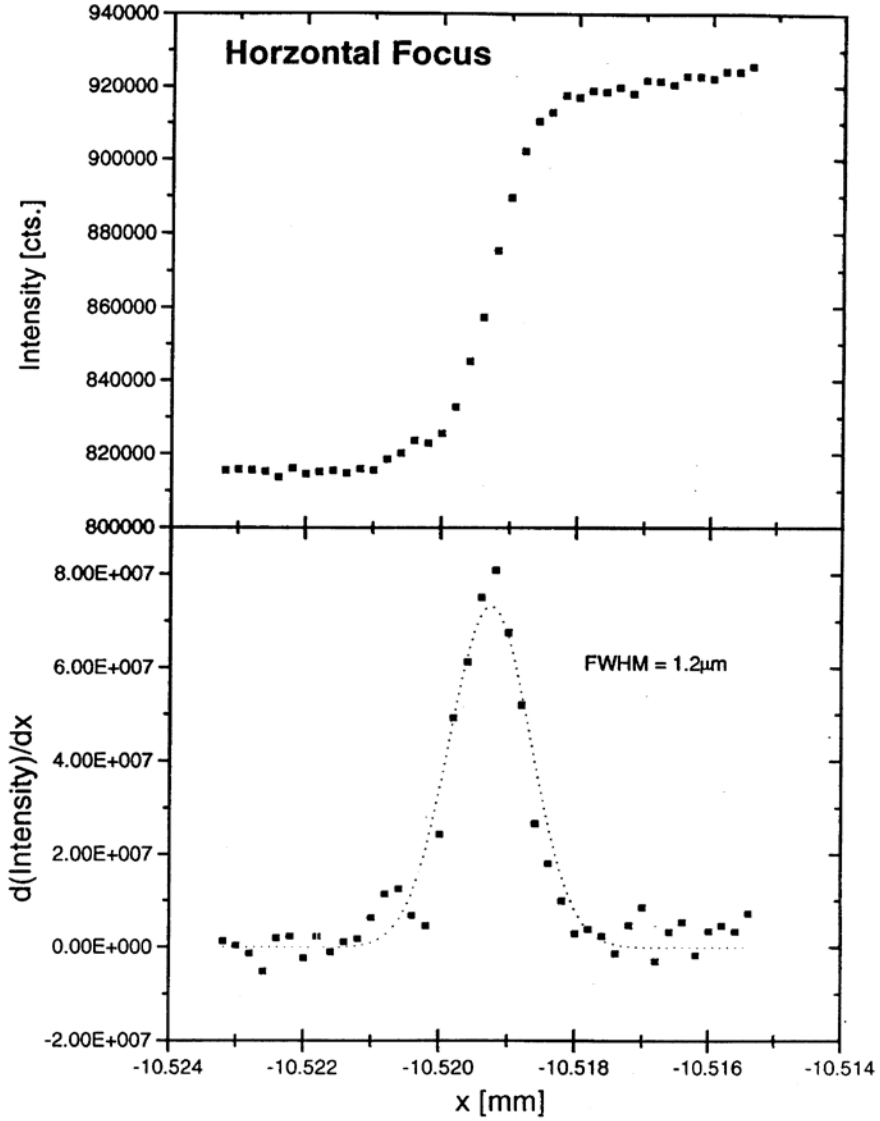
2. The age-hardening temperature to produce condition H900 (480° C) is sufficient to produce a substantial degree of stress relief.

3. The low carbon reduces the chance of retained austenite after quenching and diminishes the importance of carbide precipitation and carbon migration processes.

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. The larger lattice distortion associated with the interstitial position of carbon appears to be the main reason why it plays such an important role in dimensional stability and why its reduction or elimination is so beneficial for such stability.

5. 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. Thus low carbon is important to dimensional stability and =17-4 PH S/S has low carbon

OPTICAL PROFILER TRACE OF S/S MIRROR



X-RAY SPOT SIZE OF S/S MIRROR