NICKEL PLATED INVAR MIRRORS FOR SYNCHROTRON-RADIATION BEAM LINES

By

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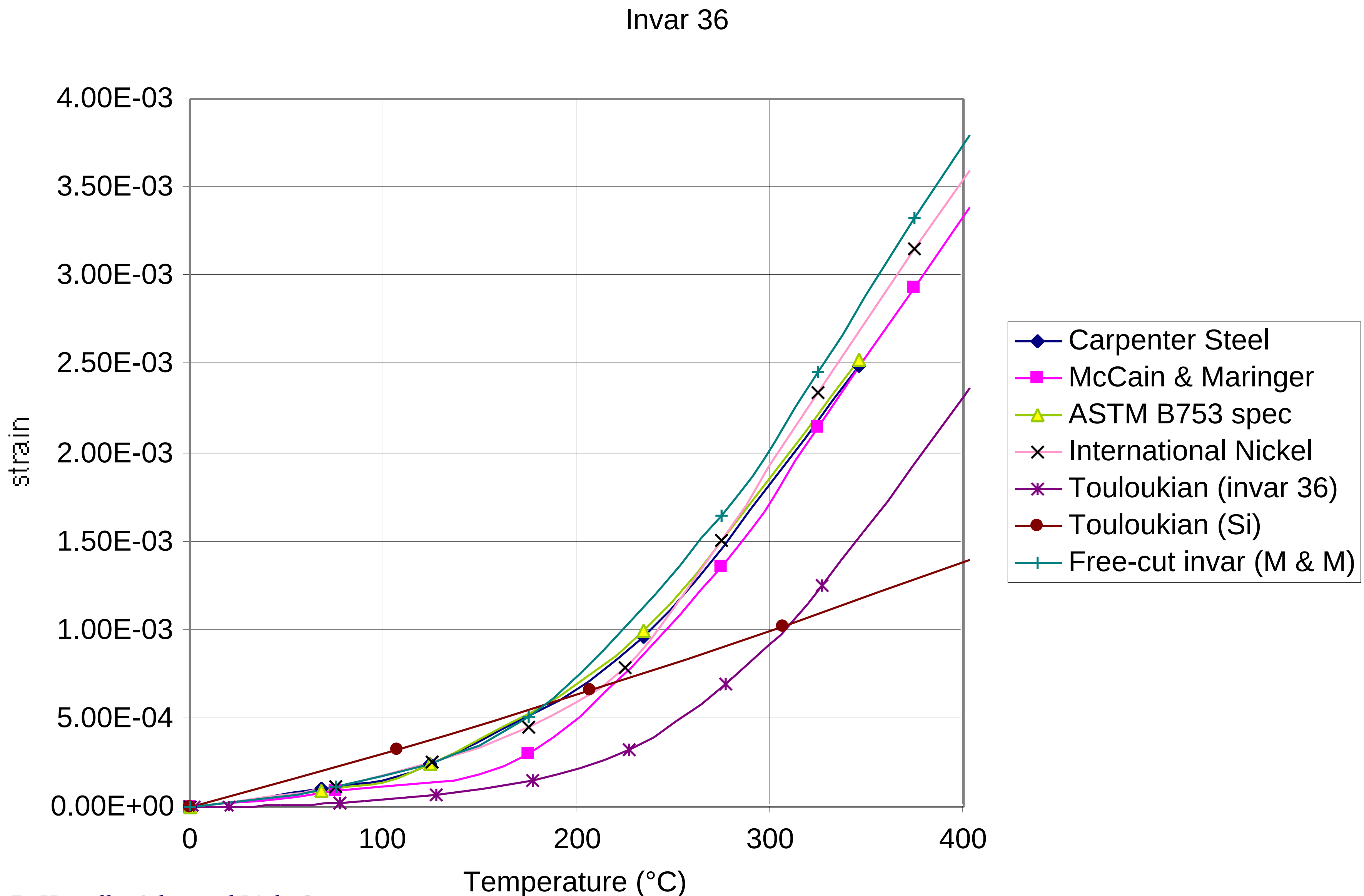
INVAR AS AN OPTICAL-SUBSTRATE MATERIAL

- Invar (36% Ni+64%Fe) has good thermal parameters compared to other metals in the temperature range 0-80°C
- It has most of the advantages of a steel alloy
  - widely available with good quality control
  - weldable, brazeable
  - machinable like other steels
  - magnetic - easy to grind
  - almost stainless
- Discovered in Paris by Charles Guillaume in 1886
  - it is an austenitic (fcc) material which is ferromagnetic at room temperature with a Curie temperature of 260°C
  - the invar property results from a balance between a decrease of atomic spacing due the loss of magnetic ordering as the Curie temperature is approached and the normal increase in atomic spacing with temperature
  - the invar property is compromised by impurities especially C which should be below 0.01% and Mn
- Invar can only be used for optics if the problem of dimensional instability can be addressed - much studied by Guillaume - γ expansion - 50 ppm total over 20-30 yrs

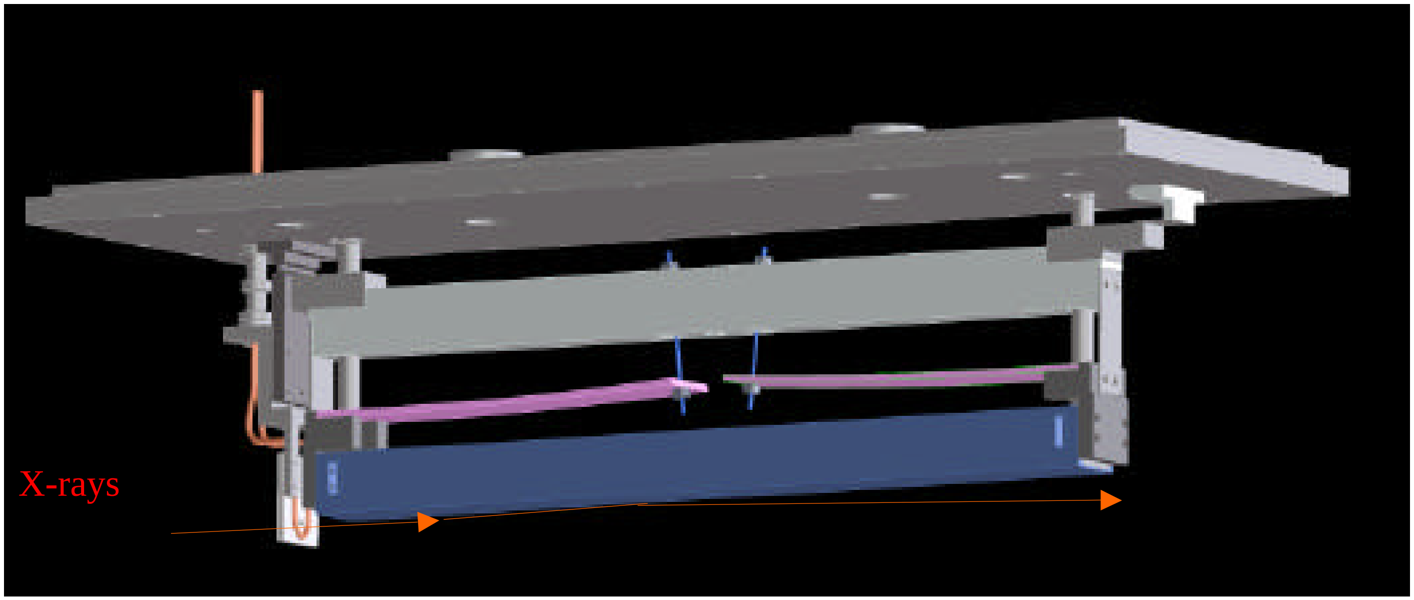
THE DIMENSIONAL STABILITY ISSUE

- The γ expansion was first understood in a landmark paper by Lement, Averbach and Cohen in 1951 - the problems were recognized to be:
- A. Stress relief
  B. The γ expansion
  C. Graphite formation
- The solution for best compromise between dimensional stabilty and low expansion coefficient is “The MIT triple treatment”
  1. Solution treatment: heat to 830°C for 0.5 hour, quench (polyalkylene glycol is best)
  2. Stress relief: heat to 315°C for 1 hour in air, air cool
  3. Stabilization: heat to 95°C in air for 48 hours, air cool
- The stabilization is intended to get the γ expansion completed quickly
- Step 2 can be repeated as often as needed during manufacture but after stabilization the part must be held below 90°C thereafter
- The expansion coefficient obtainable with low C after this is around 1 ppm/°C

INVAR THERMAL EXPANSION DATA

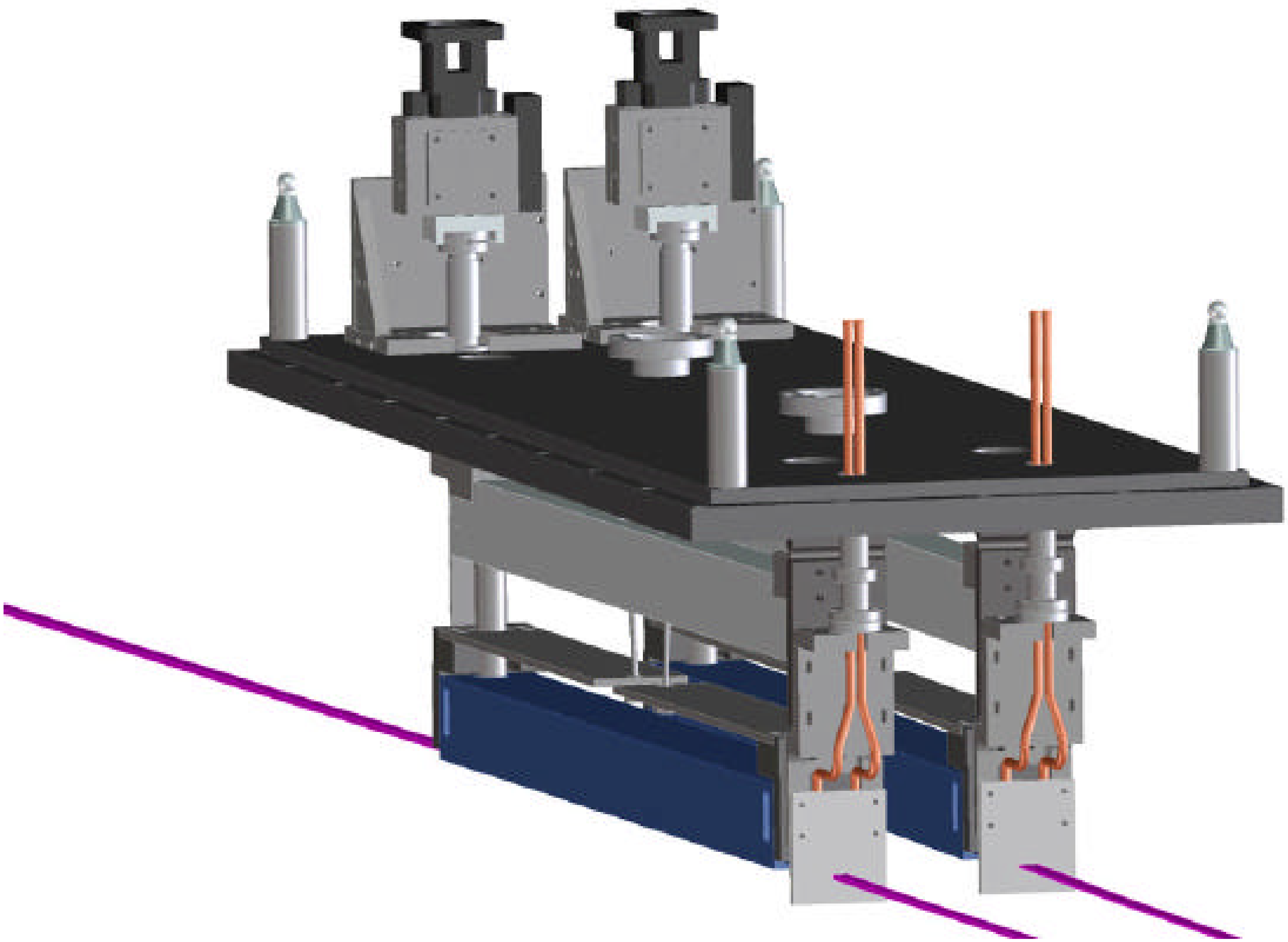


INVAR PX MIRROR DESIGN CONCEPT

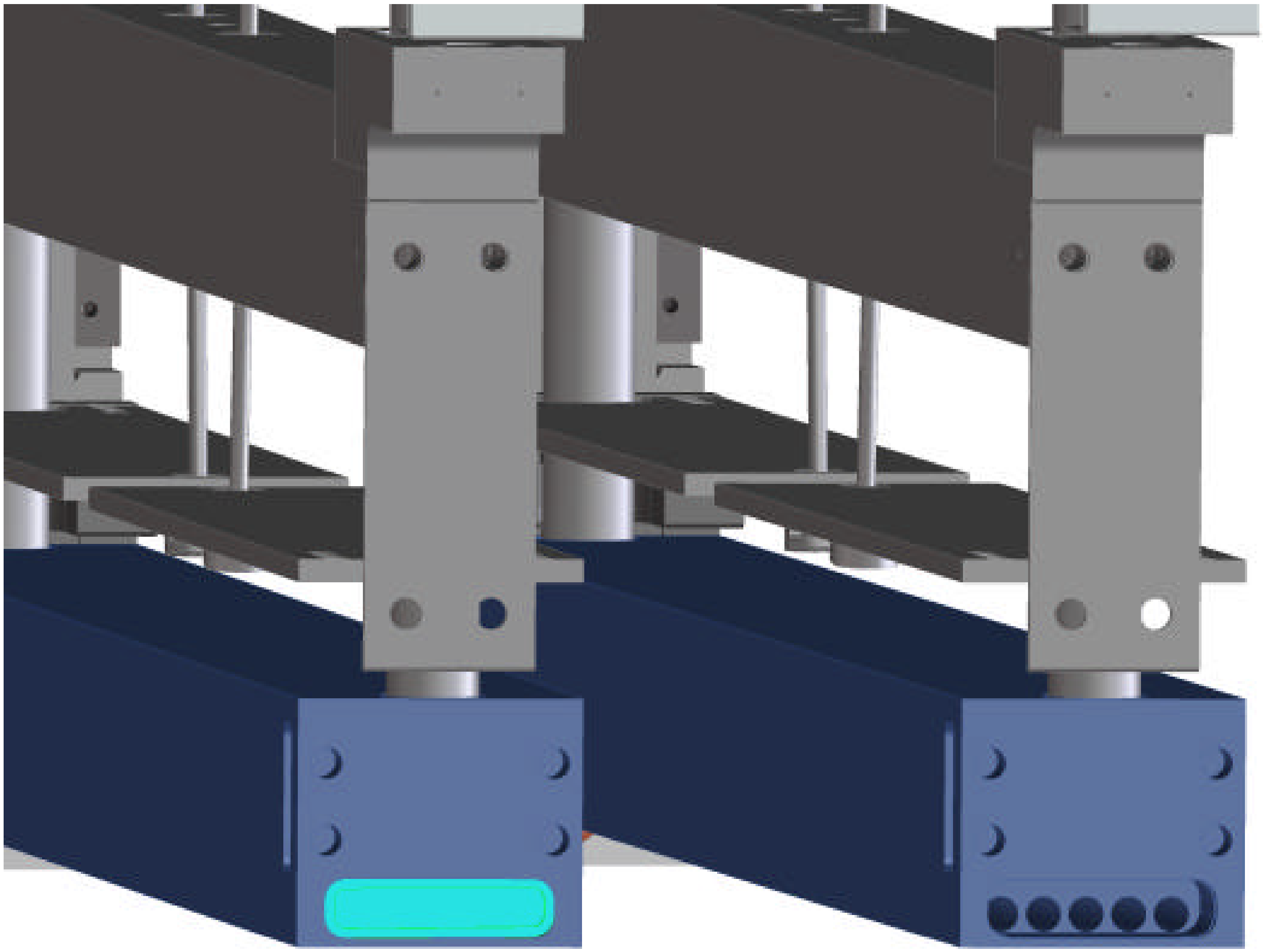


- Reflecting surface faces downward
- Bending forces are applied by adjustable flexible leaf springs
- Mirror shape is a cubic approximation to the ideal parabolic cylinder that collimates the beam for entry into the double crystal monochromator

MIRROR PAIR TO BE 6.5 m FROM SUPERBEND



MIRROR PAIR CLOSE UP



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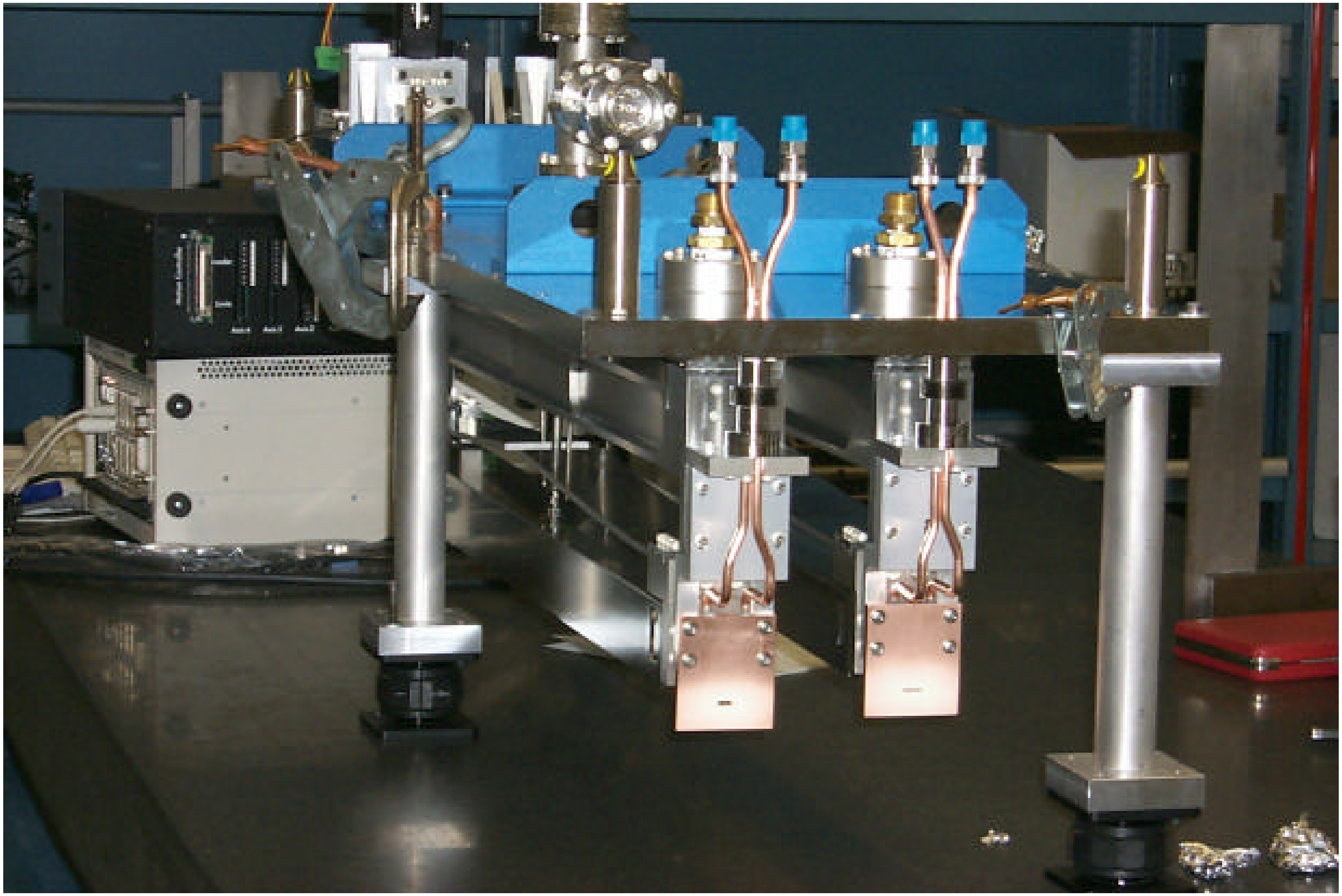
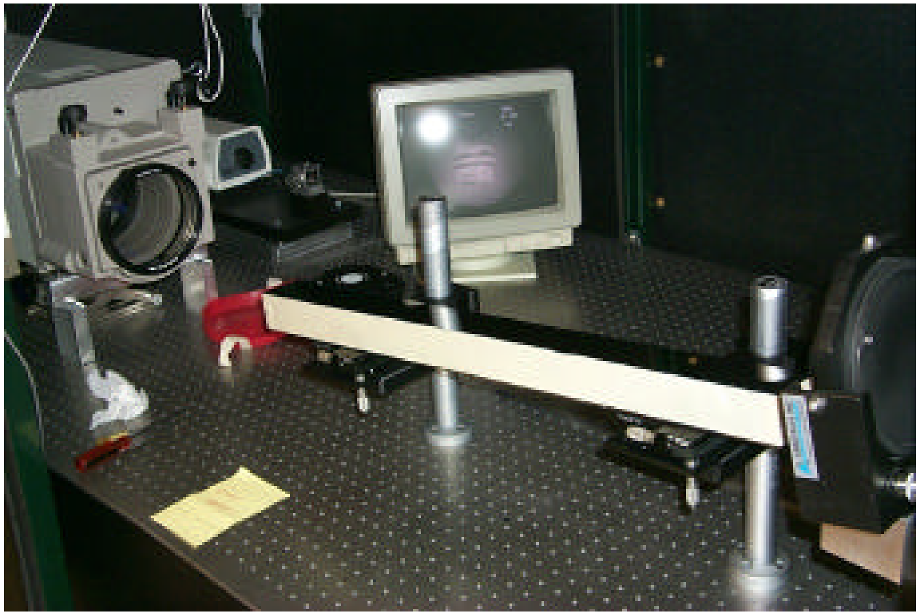
MIRROR PAIR DURING ALIGNMENT



MEASUREMENTS OF MIRROR QUALITY



An invar mirror undergoes interferometric testing for figure accuracy at the ALS Optical Metrology Lab

- First four mirrors were measured for figure and finish
- Measurements made with no gravity effect (reflecting surface vertical)
- Figure spec was 2 μr RMS after removal of piston, tilt and power
- Results in μr RMS:1.26, 2.62, 1.60, 1.52, Mirror absent: 1.50 - which shows that the mirrors are mostly close to 1 μr RMS
- Finish results less good - around 15 Å - not so critical

MEASUREMENTS ON NINE INVAR MIRRORS

MIRROR NAME	BL	BLISTERS NOW?	POLISHER MEASUREMENTS		ALS MEASUREMENTS				
			Finish (Å rms)	Slope (μr)	Finish (Å rms)	Slope (μr) GPI 2000	Slope (μr) GPI 2001	Slope (μr) LTP 2000	Slope (μr) LTP 2001
SBM101		Yes many	4.9	0.76	7.0		4.5		7.5
SBM102		Yes many	4.8	0.90	7.4		5.9		4.5
SBM103		Yes many	4.3	1.00	6.5		2.9		4.0
SBM104		No	5.2	0.97	13.6		2.0		2.6
SBM105		No	5.1	1.5	23.1		2.0		2.1
SBM106	8.2.1	1 big just outside CA*	6.3	0.20	13.2	1.3	1.54	1.9	
SBM107	8.2.2	1 big in CA*	8.4	0.24	17.2	1.6	2.26		
SBM108	8.3.1	No		0.22	14.6	1.5			
SBM109	None (uncooled)	No		0.35	15.1	2.6	2.4	3.3	2.9

*These were not seen in the measurements a year earlier (CA=clear aperture), [#]BL=beam line

BLISTERING PROBLEM

Conclusions after Paquin consultation:

- Blisters due to separation of plating layer
- Cause is unsuitable or poorly executed preplating cleaning and preparation
- Limited information from plater seems to confirm poor QA
  - Lack of starter process: no electrolytic strike
  - Lack of stress monitoring
  - Phosphorus content not well monitored but probably too low
- "Processes specified for the fabrication of these mirrors are sound and should be implemented with minor changes"
- We have suffered quality control problems and should seek solutions via improved quality control
- By this time we already had a successful Ni-plated invar mirror in hand (the BL 7 grating substrate) from InSync/Acteron and we decided to use these vendors but carry out a program of tests on Acteron-plated invar

NICKEL-PLATED-INVAR TEST PROGRAM

Stripping test:

Coupons machined on all sides then lapped on one side - plate with 4 mils all over except only 2 mils on the lapped side - question is will the stripper make pits in the invar on the thinly plated face

Stress test:

Strips are plated on only one side, the curvature indicates the final stress (Acteron also uses a standardized in-bath stress monitor)

Adhesion test:

Prescribed in the MIL-SPEC and ASTM spec the test is to plate a strip on both sides and then bend it 180° around a mandrel of diameter four times the strip thickness (10 mm minimum) - the plating is allowed to crack but it must not lift off even when prised with a sharp blade

Bakeout temperature test:

The last two tests are to be performed for two bakeout regimes (a) 275°C for 1 hour and (b) 225°C for 2 hours to test if there is any important effect on stress or adhesion. This is a general concern not special to invar

ELECTROLESS NICKEL GENERAL PROPERTIES

- Electrless nickel is a chemically-applied coating that is applied mostly to metals to gain corrosion and wear resitance - it is now a huge industry since its commercial beginnings in the 1950's - ASM Handbook lists 140 applications and hardly mentions optics!
- Provides a superpolishable surface for essentially any metal substrate
- Alloy of nickel and 5-13% phosphorus
- For SR substrates (other than Al), 9-11% is normally used which gives the desired hard amorphous coating
- Propietary solutions contain Ni and P ions and a reducing agent (usually sodium hypophosphite) plus various control additives
- The Ni and P concentration and the temperature and pH are normally monitored: T = 90°C and pH = 4 to 5 are typical values
- For optics a coating of about 100 μm is typical which takes about 10-40 hours and is intended to allow for sufficient material removal during polishing - note this implies a flatness requirement!

PLATING ISSUES FOR INVAR MIRRORS

- Electroless nickel plating issues represent the main difference between making an optic out of invar compared to any other type of steel
- The main issue is stress which is always present for three reasons
  1, intrinsic depending on %P and deposition parameters
  2. CTE missmatch on cooling bath to room temperature
  3. shrinkage on bakeout
- The problem is similar to plating Al which has an equally big thermal missmatch with nickel but in the opposite direction - for invar the nickel will be in tension, for Al in compression
- In practice it is not easy to get an exact balance among the above stress factors nor is it necessary. Measured values of the stress show a wide variation and 10-30 ksi is not unusual even for materials that appear to have CTE's that are well-matched to nickel

STRESS MEASUREMENT

$$\text{STRESS} = \frac{4}{3} E_2 \left(\frac{t^2 + td}{d\,\ell^2} \right) \Delta L$$

E_2 – YOUNG'S MODULUS BASIS METAL

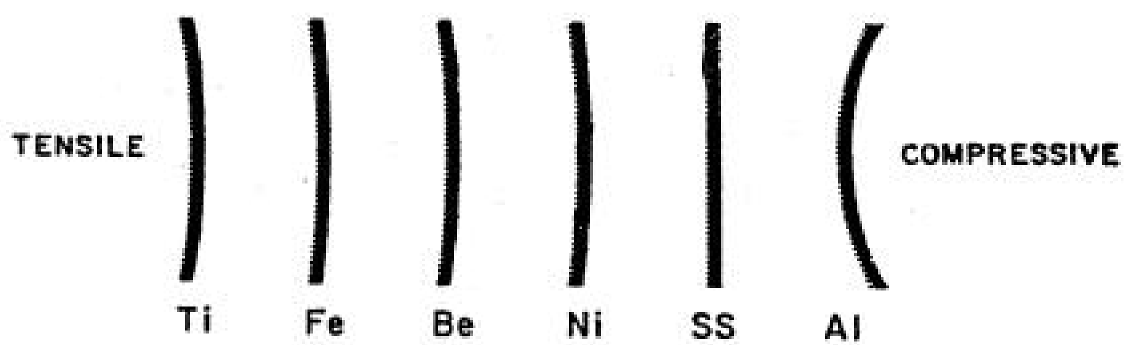
d – DEPOSIT THICKNESS

t – STRIP THICKNESS

ℓ – LENGTH OF PLATED STRIP

ΔL – NET CHANGE IN BOW AT CENTER OF STRIP

Stoney Equation (1909)



- Standard to use witness strips with every plating bath
- Invar would be to the left of Ti on the bottom diagram
- Can also use optical measurements of the curvature of actual mirrors - eg BL 7 grating substrate gives 20 ksi

STRESS IN ELECTROLESS NICKEL PLATED LAYERS

K. Parker 1981

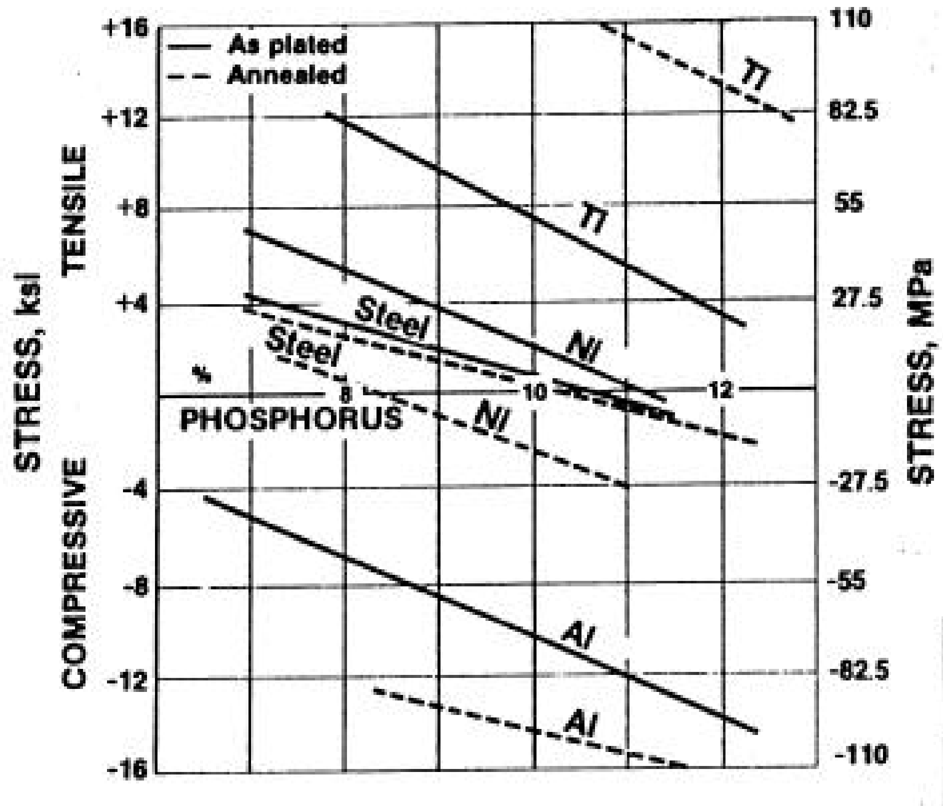
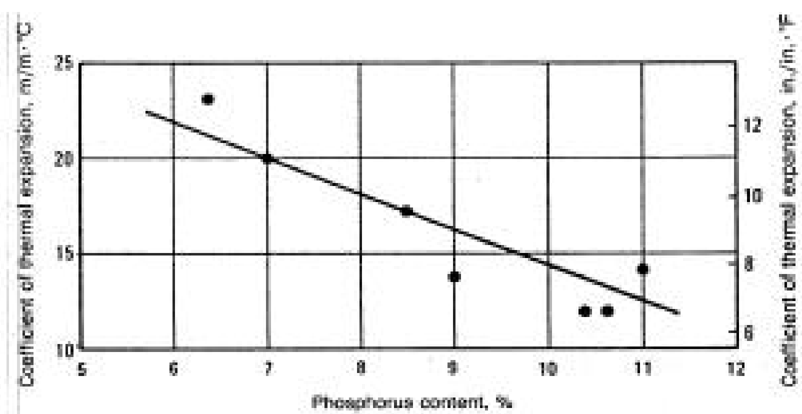


Fig. 6—Stress of 25-μm-thick electroless nickel deposits.

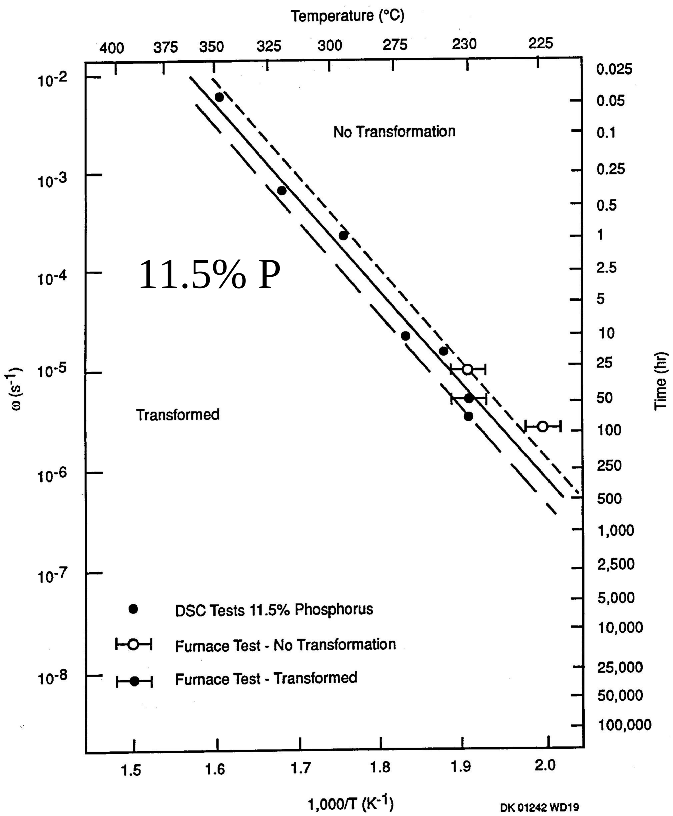
Mat'l	CTE (ppm/°C)
Invar	1.0
Ti	8.35
Steel	12.2
Ni	13.3
Al	22.5

- Diagram shows the general trend toward lower tensile stresses at higher %P
- We see why we are at around 12%
- Invar would be roughly at the blue lines which makes our observed stress of 30 psi on test strips seem quite reasonable

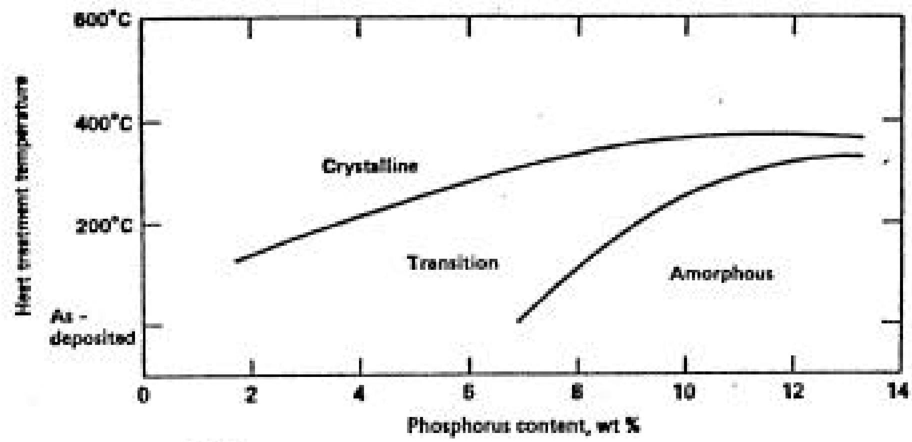


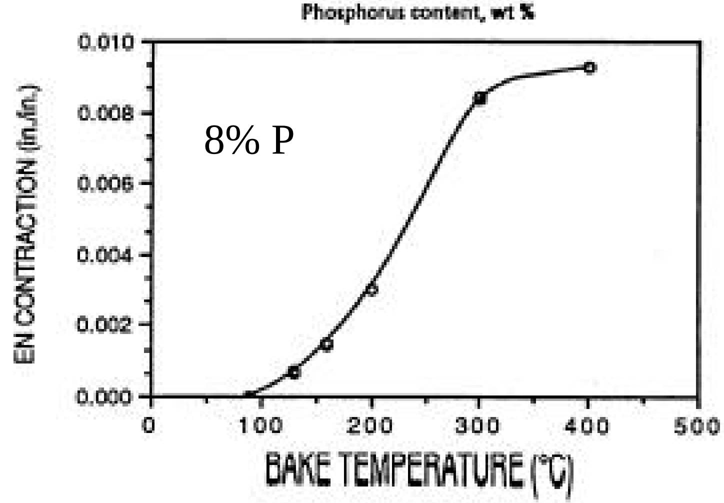
BAKING ISSUES FOR ELECTROLESS NICKEL

- A roughly 200°C bake is usually done for the following reasons
  - remove hydrogen and (in some cases) prevent embrittlement
  - improve adhesion
  - increase hardness (good for polishing)



- There is a phase transition to $Ni+Ni_3P$: high shrinkage, loss of amorphous character and worse polishability
- Therefore limit bake time and temperature to around 200°C - 300°C too high





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TEST RESULTS

- **Stripping test**
  The Ni was stripped effectively without damage to the invar surface, even for thinly plated areas that had long exposure to the stripper - (100% of 4 test coupons)

- **Stress test**
  Low-T bake (4 strips): average curvature change = 0.75 m
  High-T bake (4 strips): average curvature change = 0.82 m
  These results are similar and indicate a stress level of about 30 ksi - BL 7 grating has 20 ksi - not unusual even for substrates with a good CTE match to nickel

- **Adhesion test**
  All strips passed (100% of 8 strips; 4 high-T bake, 4 low-T bake)

- **Bake-temperature test**
  Low-T bake (3 disks):
  High-T bake (3 disks):
  All polished to 7Å rms with no observable difference between high and low temperature bake

SUMMARY

- We have nine invar mirrors designed to be collimators PX beam lines
  - One is uncooled and not used
  - One is held as a spare
  - Three are feeding PX beam lines (with 100 micron collimators) which suffer a 2-5 times lower than expected flux largely due to the mirror imperfections
  - Four have been stripped and are being plated and polished by InSync/Acteron
- In view of the tests and the BL 7 optic we expect good results
- The outcome of this process should be available in time for the manuscript of this paper