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10.6 MICROMETER ABSORPTION IN MOLYBDENUM MIRRORS

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July 1974

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AIAA/ASME 1974 Thermophysics
and Heat Transfer Conference
Boston, Massachusetts

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10.6 μm Absorption in Molybdenum Mirrors

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January 1974

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ABSTRACT

Absorption of laser light is a fundamental and important mechanism of heat transfer into molybdenum which is used extensively in high intensity lasers. This paper discusses properties of molybdenum and the effects of surface finish on the absorption of bare, silver coated, and gold coated molybdenum mirrors. Mirror characterization includes 10.6 μm absorption and/or reflectivity, surface roughness, phase-contrast microscopy, pulse and preliminary CW laser damage studies. Interferometric versus stylus instrument measurement of surface roughness is discussed. The polishing and coating procedures are described. Some limited characterization of high intensity laser operational mirrors is also included.

Keywords: Absorption, Molybdenum, Laser Mirrors, Reflectivity, Surface Roughness, Laser Damage, Metal Polishing, Metal Coating

10.6 μm ABSORPTION IN MOLYBDENUM MIRRORS

I. INTRODUCTION

Absorption of laser light is a fundamental and important mechanism of heat transfer into molybdenum which is used extensively in water cooled mirrors for high intensity lasers. Goals of high intensity laser mirror development include the minimizing of the absorption so as to reduce thermal distortion and decrease the probability of laser damage. This paper will discuss properties of molybdenum and a study performed to evaluate the effects of surface finish on the absorption of bare, silver coated, and gold coated molybdenum mirrors. Some absorption data from operational molybdenum mirrors and limited laser damage results will also be included.

II. THE CHOICE OF MOLYBDENUM

Molybdenum has been chosen for water-cooled mirrors as a consequence of its thermal and mechanical properties and the following considerations. A thin face plate is desired so that heat can be quickly transferred and exchanged with the water, or other cooling liquid. The thermal diffusivity is, therefore, an important parameter. The material must be strong enough not to distort exceedingly from the necessary pressure and flow rates of the coolant. Also it is desirable to have a low thermal-linear

expansion coefficient so that thermal distortion may be minimized. The properties of molybdenum are summarized in Table 1.

Most molybdenum used in high power laser mirrors is ASME 7805 with low carbon exception, vacuum arc cast. The major impurity of carbon may be found as sub-micron (about 0.5 μm diameter) carbide inclusions. Carbide and other inclusions create scratches when they are lifted out during the polishing process.[4] Inclusion effects are shown in Figure 1 which is a scanning electron microscope (SEM) microphotograph of polished molybdenum. The mirrors of this study were cut from the same bar of 7305 molybdenum to be approximately 2 cm in diameter and 7 mm in thickness.

III. MIRROR CHARACTERIZATIONS

Several characterizations of the mirrors were performed. The absorption was measured directly by using calorimetric techniques at AFWL.[5] Measurements were made with a CO_2 laser operating at 15 watts. The in-situ mirror heat capacities were measured using an electrical loading of approximately the same power and energy as the laser, and monitoring the temperature rise. The precision of the device is estimated at 0.0002 from repeated (after removing and replacing the mirror) measurements of the some super-smooth (less than 30 Å rms roughness) samples. We attempted to locate the 6 mm diameter laser beam on center for each measurement, and, therefore, this experiment is also some

TABLE 1

MOLYBDENUM

PHYSICAL PROPERTIES

DENSITY[1] 10.2 G/cm^3 MELTING POINT[1] 2895°C

	0°C	100°C	500°C
SPECIFIC HEAT[1] J/gm°C	0.24 (0.27)[a]	0.26	0.28
THERMAL CONDUCTIVITY[1] J/sec cm°C	1.4	1.3	1.2
THERMAL DIFFUSIVITY[1] cm^2/sec	0.57	0.55	0.38
THERMAL LINEAR EXPANSION[2] %/°C	4.9×10^{-6}	5×10^{-6}	
YIELD STRENGTH[3] 1000 PSI	100	70	--
VICKERS HARDNESS[3]	--	240	180
TENSILE STRENGTH[3] 1000 psi	100	95	51

a. Our value of specific heat of 7805 molybdenum used in this study.

measure of the uniformity of the reflectivity near the mirror center. Due to the nature of the systematic errors involved with our calorimetric absorption measurements, the accuracy of the absorption, A, is ±0.1A.

The reflectivity of the mirrors at CO_2 laser wavelengths was measured on our multiple-bounce reflectometer[6] which has an accuracy of 0.002 and a precision of 0.001 for this set of measurements unless otherwise stated. Conservation of energy implies a relationship between the reflectivity, absorption, transmission, and scattering of a surface given in (1):

$$1 = R + A + T + S. \tag{1}$$

Since the transmission of metal mirrors is zero, the relationship between the absorption, reflectivity and scattering can be expressed,

$$1 - R = A + S. \tag{2}$$

Since the scattering is usually small, (2) becomes

$$1 - R \geq A. \tag{3}$$

The good agreement between absorption reflectivity measurements will be discussed later.

The surface roughness of the mirrors was measured with a Model 1, Taylor Hobson, Tally Step. The Tally Step works on the principle of magnifying (up to 10^6:1) the motion of a diamond tool which travels 2.5 mm across the surface. All our measurements were made with a 13 μm radius tool. In order to investigate the effect of tool radius a measurement was made with a 1300Å radius tool with no noticeable change in the frequency or heights of the peaks, indicating

that the instrumental function had negligible effects on the recorded surface contour. Figure 2 presents contours of two mirrors. Mirror M-109 had the highest reflectivity whereas M-106 had one of the lowest. In M-106 there are areas which are smoother than M-109, but there are also large spikes. M-106 and M-109 illustrate that the RMS roughness is not an adequate parameter to completely describe the surface.[7] Another important parameter is the autocorrelation function.[8] Repeated digitization of the same 1 mm of a mirror on the same chart indicated a precision of 10Å and 25Å for 500KX and 200KX magnification respectively. In order to approximate the accuracy of our process, we digitized a FECO (Fringes of Equal Chromatic Order) interferogram taken by Dr. J. Bennett.[7] We got a roughness of 53 Å RMS compared to her value of 43 Å RMS. We feel her answer is more accurate because we took 167 points compared to her 512 points. Our interval for digitizing corresponds to approximately 0.05 mm on the mirror surface. Depending on the portion of the chart digitized and where on the mirror the Tally Step measurement was made resulted in a variation in 40Å. No compensation was made for variation in the mean surface height, except for digitizing in a portion of the scan where the mean surface height was approximately constant to 100Å. The estimated accuracy of the roughness measurement is ±60Å.

It is interesting to make comparisons between Tally Step and FECO surface characterizations. The Tally Step

using a 1300Å radius tool has better resolution across the surface of the mirror than FECO but is much inferior for resolution into the mirror surface. Observation with a 5X eye loop did not detect a scratch left by the Tally Step on our mirrors, but this may be due to the quality of the surface finish on the mirror. The Tally Surf, which is a device similar to the Tally Step, left a visible scratch on the mirror. Since the Tally Step does not require silvering of the surface, the Tally Step is the least destructive of the methods. A Tally Step measurement takes about 2 minutes after a 5 minute set up. Comparisons between surface characterizations using FECO, Tally Step, and light scattering should be interesting and may yield insight into the complex picture of effects of surfaces and high power laser mirror performance.

IV. MIRROR PREPARATION

The mirrors were ground and polished simultaneously in one block as described in Table 2. It is significant to note that only Al_2O_3 was utilized for an abrasive. The type of pitch seems to be important. Perkin-Elmer has had extreme success in using Swiss pitch in polishing molybdenum, beryllium-copper, copper, and TZM.

Some of the mirrors were selected for coating with chrome-silver-thorium-tetrafluoride, or chrome-gold. The coating conditions are reported in Table 3. The samples were rotated at 12 rpm during the coating, in order to

TABLE 2

MOLYBDENUM GRINDING AND POLISHING

ABRASIVE	DIAMETER	TIME	PRESSURE
$Al_2O_3 + H_2O$	100μ	5 hrs	-
$Al_2O_3 + H_2O$	30μ	5 hrs	-
$Al_2O_3 + H_2O$	9μ	3 hrs	-
H_2O + LINDE A (Al_2O_3)	0.3μ	10 hrs	20 psi
H_2O + LINDE B (Al_2O_3)	0.05μ	15 hrs	20 psi

TYPE 40 (MEDIUM) PITCH

TABLE 3

COATING CONDITIONS

	SILVER	GOLD
PREPARATION	GLOW DISCHARGE	GLOW DISCHARGE
SUBSTRATE TEMP	120°C	120°C
CHROME (PRESSURE) 35% T @ 5400Å	5 Min (9×10^{-6})	4 Min (1×10^{-5})
TIME BETWEEN NEXT LAYER	5 Sec	9 Sec
PRIMARY COAT (PRESSURE)	12 Sec (3×10^{-6})	20 Sec (1×10^{-5})
ThF_4	12 Min (6×10^{-6})	N/A

improve coating uniformity. The time and pressure in the chamber for deposition are critical, especially for the case of silver. UHV (10^{-6} to 10^{-9} torr) conditions result in the highest 10.6 μm reflectivity of 0.9953 and 0.9939 for silver and gold respectively on super-smooth quartz.[9] Quick deposition times (8-12 sec) for 1000Å of silver can yield reflectivities of greater than 0.991 on smooth metal substrates even though UHV conditions are not used. Chrome is classically used as a binder for silver on glass coatings. Metal samples silver coated in our lab using a chrome binder pass the scotch tape test. Thorium-tetrafluoride is added over the silver to reduce atmospheric degradation of the silver coating.

V. RESULTS

The reflectivity, selected absorption, and the calculated RMS roughness of the molybdenum mirrors of this study are shown in Table 4. The absorption and reflectivity are in good agreement except for M-103 which was contaminated with thermal grease before the reflectivity measurement. The initial reflectivity measurement of 0.968 was raised to 0.974 by cleaning with ethanol, and it is suspected that a residual film caused the discrepancy between the absorption and reflectivity values. Note the much rougher M-106 and M-108 have a significantly lower reflectivity for the

TABLE 4

SUMMARY

MOLYBDENUM OPTICAL PROPERTIES

UNCOATED R	1-A	δ (RMS Å)	Au	Ag	#
0.880 ± .004				.980	M-113
0.925		289	0.991		M-106
0.925	0.928	284		0.989	M-108
0.971		132	0.990		M-107
0.974	0.979	123			M-103
0.974		136		0.988	M-105
0.975		137	0.991		M-112
0.977	0.979	133			M-104
0.978	0.979	180 (140)		0.992	M-111
0.979	0.979	184	0.990		M-110
0.982		150 (190)		0.990	M-109

uncoated case. The roughness values in parentheses indicate a value obtained by digitizing a different section or a different Tally Step run on the same mirror.

Phase-contrast (Nomarski) microscopic investigations were made of the surface. Figure 3 presents Nomarski photographs of mirrors M-107 through M-109. M-108 had one of the lowest reflectivities of the mirrors and exhibits a minimum area of smoothness and the greatest densities of black spots. Comparisons between molybdenum ground with diamond and Al_2O_3 indicate that these black spots are due to microgrit (Al_2O_3) which has not been removed in the polishing procedure. For this series of mirrors we found that minimizing the microgrit and maximizing smooth areas (as viewed by Nomarski) tended to maximize 10.6 μm reflectivities of the uncoated mirrors. M-113 came loose from the block and was not well polished; Figure 4 shows the very rough surface and presence of microgrit in the area where the reflectivity was measured. Figure 5 shows a molybdenum mirror which was made from the same bar as the mirrors in this test, but polished by Perkin-Elmer, Wilton, Conn. M-38 had a surface roughness of 42Å RMS as measured by FECO. The white spots on the picture are on the mirror surface. Under contract to us, Perkin-Elmer has developed a technique to polish molybdenum, TZM, and copper to less than 20Å RMS. After polishing molybdenum to 40-60Å RMS, they sputter molybdenum on top of the surface and polish the sputtered layer resulting

in surfaces typified by M-23 (20Å) RMS as shown in Figure 6. The absorption of Perkin-Elmer molybdenum mirrors from the same bar (but different polishing process than ours) and our mirrors are plotted versus roughness as shown in Figure 7. A linear regression routine has been used to plot a 2nd order polynomial through the data. Note how the reflectivity of the uncoated surface decreases for roughnesses less than 100 ± 60Å RMS. This decrease in reflectivity with decreasing roughness has been observed by others.[10] One hypothesis to explain the reflectivity degradation is that work hardening or other forms of surface damage occur during the polishing.

Coatings can decrease the absorption of mirrors. It has been found that the reflectivity of UHV deposited silver on calcium fluoride roughened quartz decreases with increasing roughness.[9] This result motivates us to improve the smoothness of our mirrors before coating. Table 4 presents the reflectivities of the coated samples. Except for M-113 which was much rougher than 300Å RMS, the coated reflectivities for both the gold and silver are about the same at 0.990. Reflectivities of microscope slide witness samples placed at about the same physical position as the mirrors yielded reflectivities of 0.990 and 0.991 for the silver and gold respectively. Decreasing the silver deposition time to 8 seconds may improve the reflectivity a few tenths of a percent. Decreasing the roughness below 20Å for molybdenum results in a reflectivity of 0.994 for a UHV silver coating.[11]

VI. OPERATIONAL MOLYBDENUM MIRRORS

Table 5 presents the results of reflectivity measurements of reflectivity of mirrors after their use in high intensity laser systems. More than one entry for a mirror indicates measurements made in more than one place on the mirror. The very low uncertainty for mirror H-11 results from repeated measurenent on the same spot. The reflectivity of a damaged portion of H-2 of 0.975 demonstrates that although a surface has a poor reflectivity in the visible the CO_2 reflectivity can be greater than 0.95. Figure 8 shows a photo of Nomarski investigation of HRL 19 near the spot which exhibited a 0.992 reflectivity. The difference in the surface topography between HRL 19 and the other molybdenum mirrors of Figures 3 through 6 may be due to HRL being made from sheet molybdenum whereas the other mirrors were made from bar stock.

VII. DAMAGE PROPERTIES

Pulsed damage studies have been performed by Soileau and Wang.[12] They used a CO_2 single transverse mode laser with an effective pulse width of 0.6 μsec focused to a spot size of $1.26 \times 10^{-3} cm^2$. They found that dispersion-hardened sputtered copper polished to 15Å RMS had the best damage threshold of 130 J/cm^2, whereas molybdenum polished to 53Å RMS had a damage threshold of 30 J/cm^2. Additional studies, with the same laser, on molybdenum polished to better than 50Å RMS and OFHC copper yielded similar damage thresholds.[14] Pulsed damage mechanisms

TABLE 5

OPERATIONAL MOLYBDENUM MIRRORS

MIRROR	COATING	REFLECTIVITY
HX	$Ag/ThF_4/CdTe$	.990
H-2 (5" Diam)	?	.982, .987
H-2 (Damaged)	?	.975
H-11	?	.9870 ± .0002, .9880 ± .0002
HRL-49	Ag/ThF_4	.988, .985
HRL-19	Ag/ThF_4	.992, .989
SOR MASTER OSCILLATOR	?	.985
ANNULAR COUPLING	?	.987

9e

in metal mirrors and causes of molybdenum's damage threshold being lower than copper are not presently well understood.

We have performed initial CW CO_2 laser damage studies with an unfocused 1 KW 971 Sylvania single-mode laser. Uncoated M-103, gold coated M-106, and silver coated M-108 were tested to 1.0 KW/cm^2 for 20 seconds with no damage observable with the Nomarski microscope at 100X. M-108 was then allowed to accumulate dust, and one half of the mirror cleaned so that the CO_2 beam was divided in half by the "dust line." After an illumination of 150 W/cm^2 and 20 seconds, laser damage was readily visible to the eye on the dust covered area.

VIII. SUMMARY AND CONCLUSION

This paper has presented the study of optical properties of 7805, low carbon exception molybdenum. The effects of surface finish on uncoated reflectivities exhibit an increase in reflectivity while decreasing the surface roughness to about 100Å RMS. Decreasing the surface roughness lower than 100Å seems to decrease the reflectivity of the uncoated molybdenum. The best 10.6 μm reflectivity for uncoated molybdenum is near 0.982.

Coating with silver or gold increases the reflectivity over 0.99. Standard vacuum coated mirrors do not show a dependence of reflectivity on roughness for roughnesses of 100 to 300Å RMS. UHV coatings on molybdenum smoother than 20Å RMS can yield a reflectivity of 0.994.

Pulse CO_2 laser damage studies show that molybdenum has a significantly lower damage threshold than copper. Initial CW CO_2 laser damage results indicate a damage threshold greater than 1 KW/cm^2 for uncoated, silver coated, and gold coated molybdenum. Dust can lower damage thresholds appreciably to less than 150 W/cm^2.

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FIGURE CAPTIONS

1. Inclusion related scratches in molybdenum near a pulsed laser damage site.[14]

2. Portion of Tally Step data taken at 500KX magnification.

3. Nomarski microscope investigation of mirrors M-107 - M-110 inclusive.

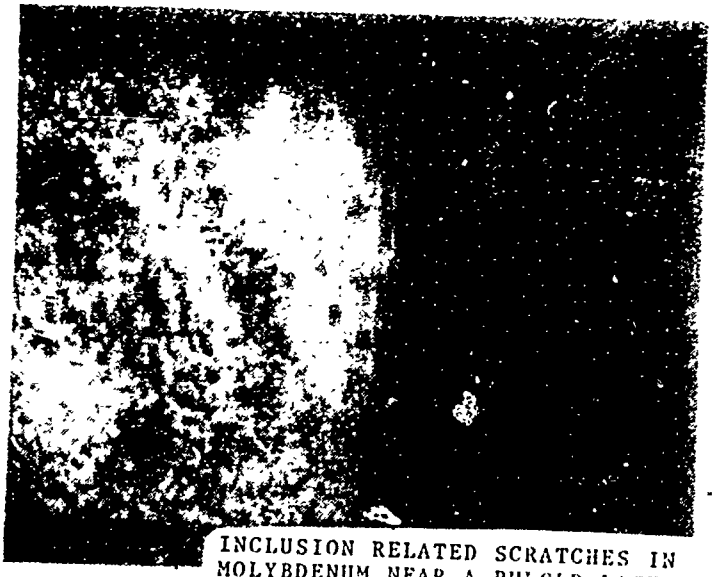
4. Nomarski microscope investigation of M-113 demonstrating presence of residual grinding compund. (Al_2O_3).

5. Nomarski microscope investigation of molybdenum mirror polished by Perkin-Elmer, to a surface finish of 42Å RMS. The white spots are on the mirror.

6. Nomarski microscope investigation of molybdenum sputtered on molybdenum and then polished by Perkin-Elmer to a 20Å RMS roughness.

7. Dependence of 10.6 μm absorption on RMS roughness for uncoated molybdenum from the same bar. The line is a second order polynomial with a peak at 100 ± 60A.

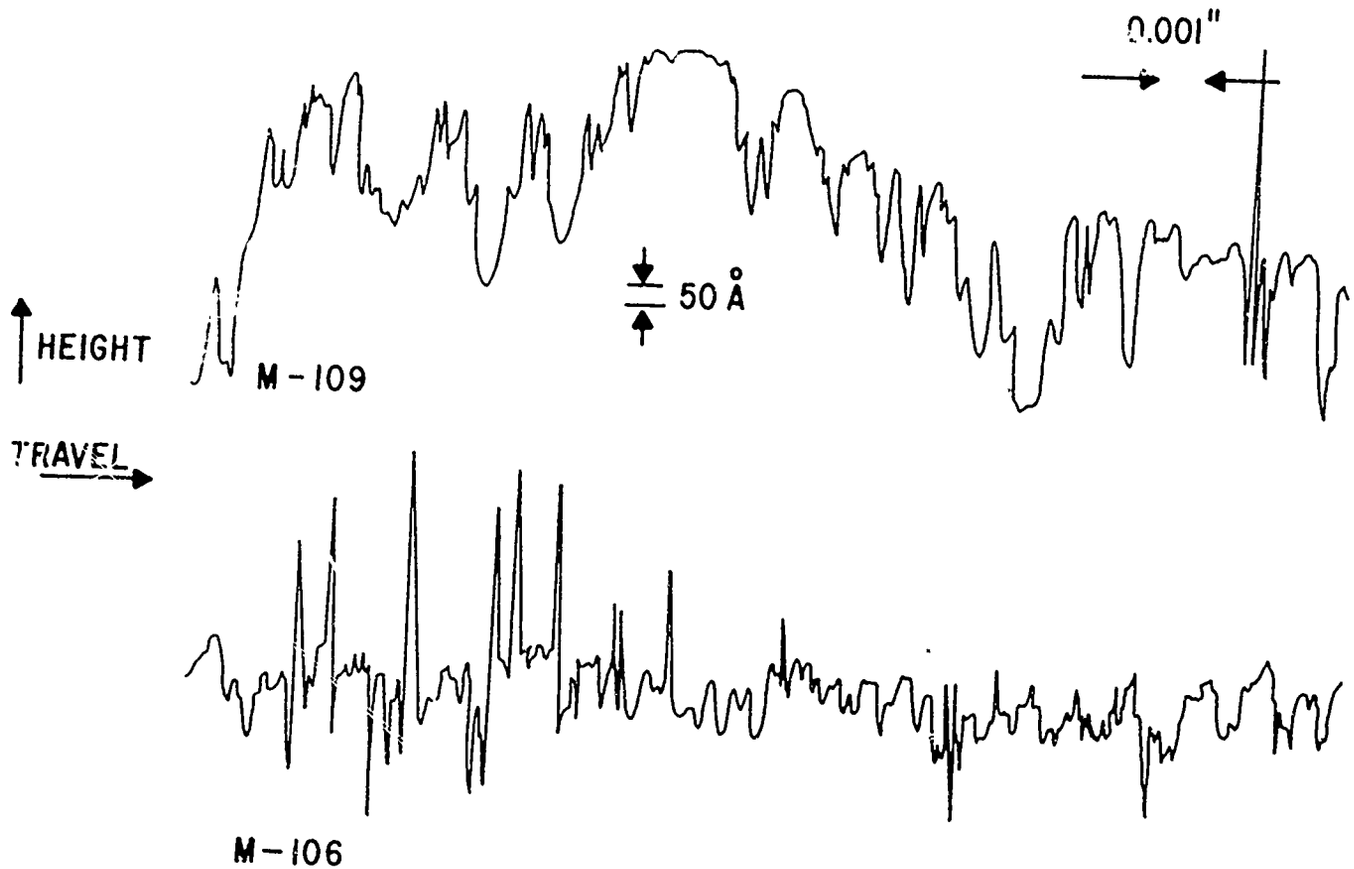
8. Nomarski microscope investigation of water-cooled operational molybdenum mirror near area which had a 10.6 μm reflectivity of 0.992. Note the different topography as compared to Figures 3, 4, 5, and 6.

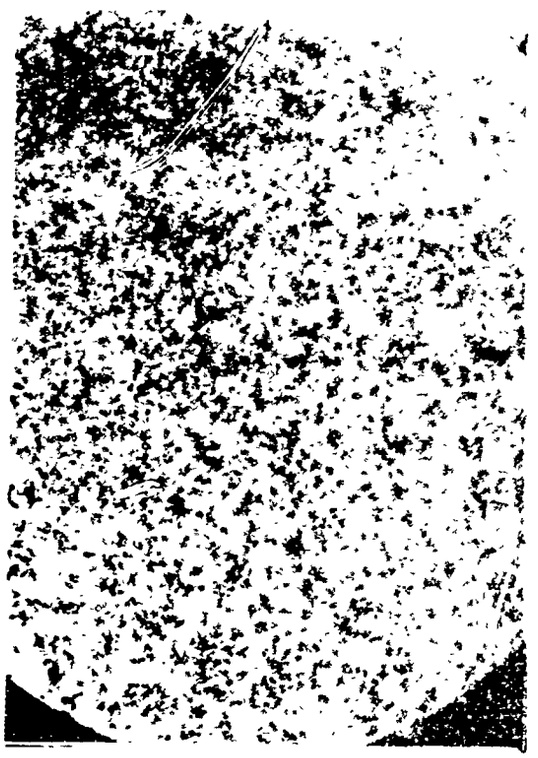


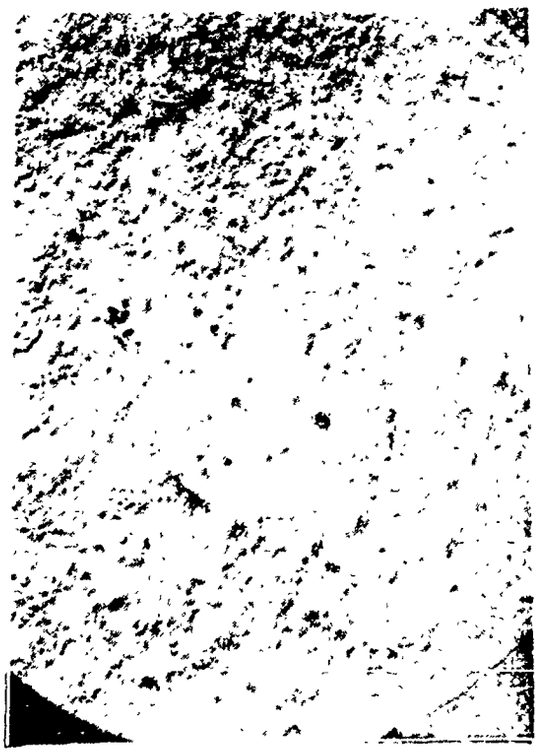
INCLUSION RELATED SCRATCHES IN MOLYBDENUM NEAR A PULSED LASER DAMAGE SITE. (SLM PHOTO AT 450 X)
10μm

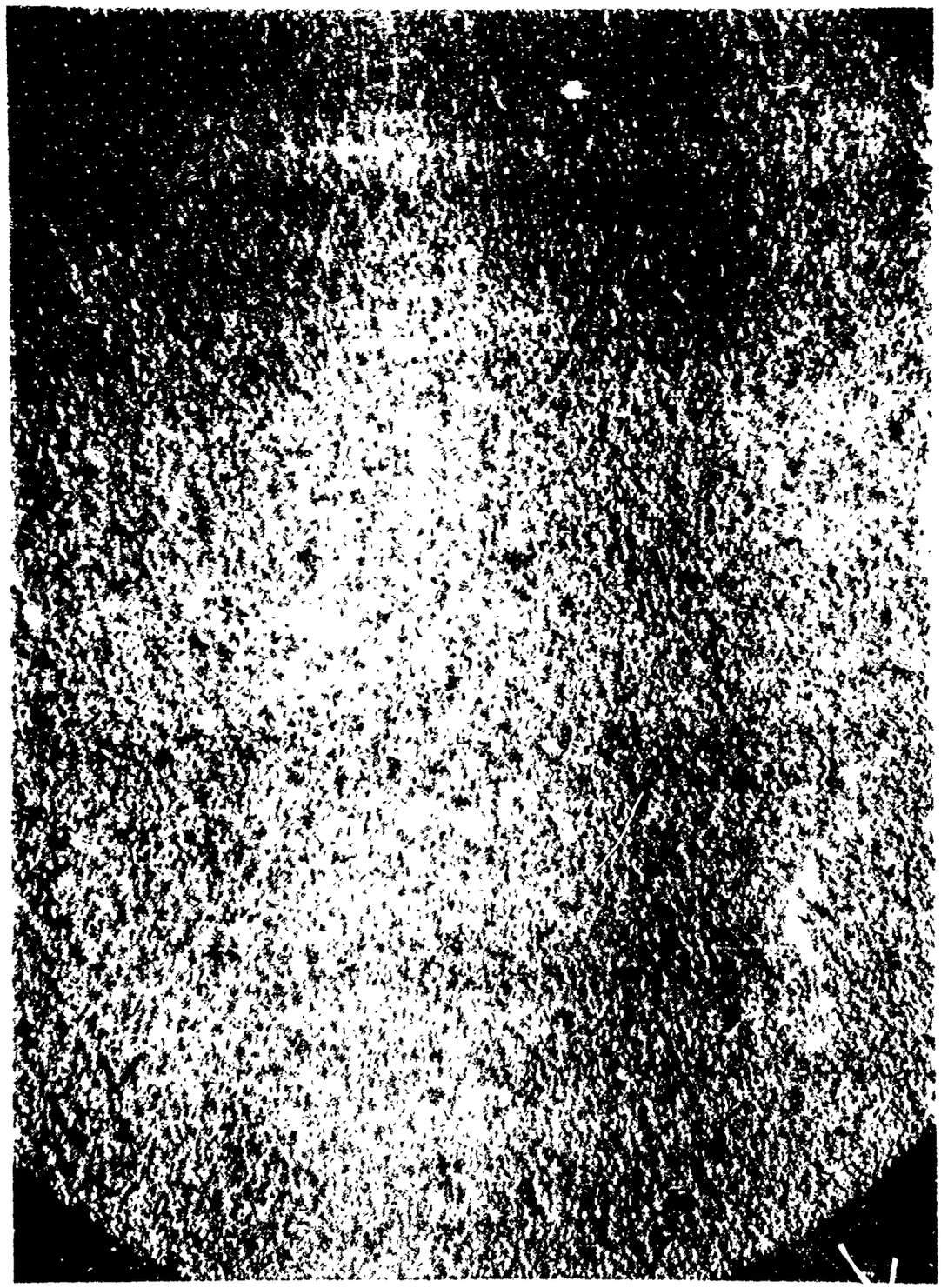
TALLY STEP DATA

500,000X



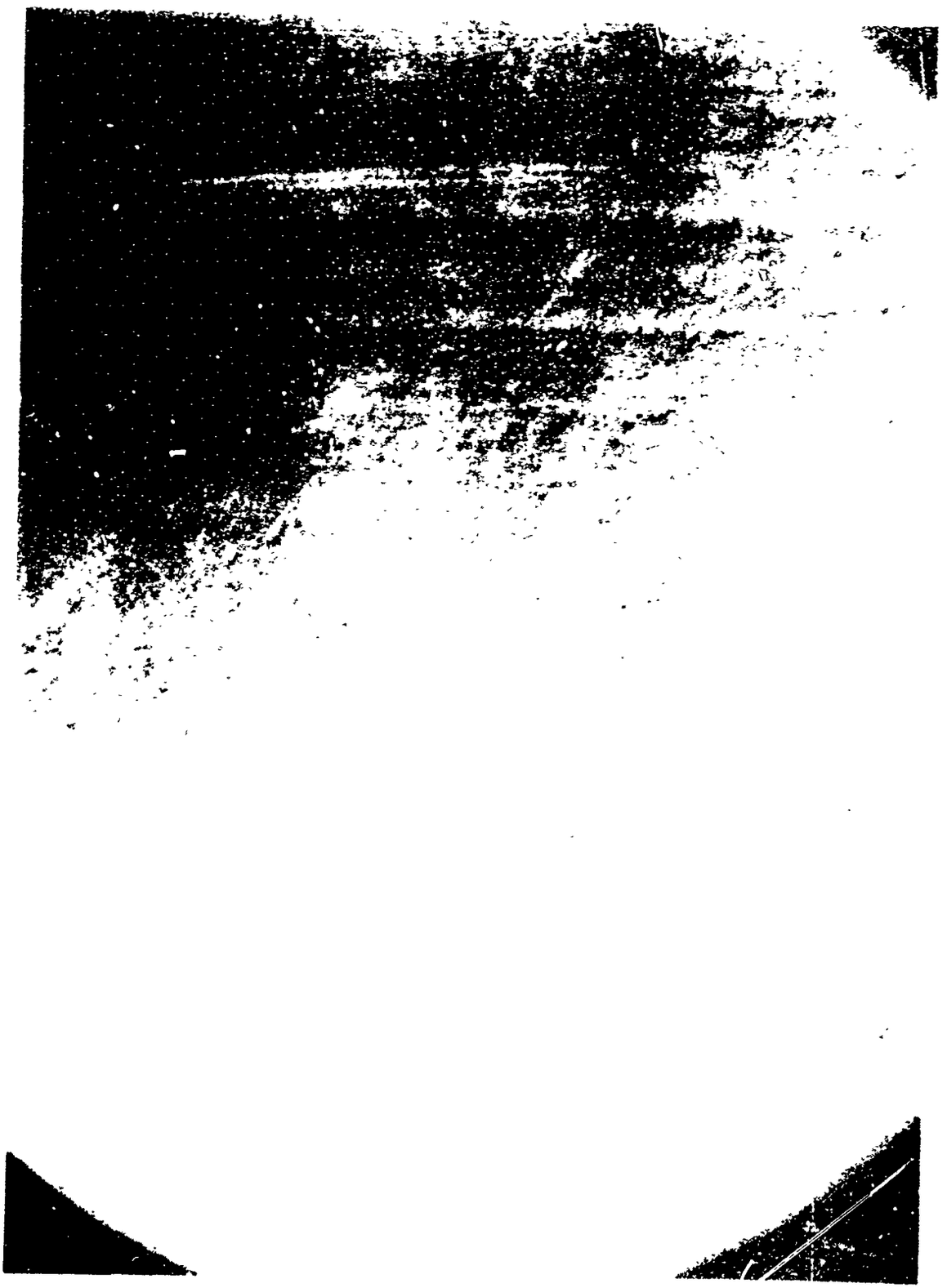






61-3 , M-113, 9[illegible]X, [illegible] Nov 73, where R measured

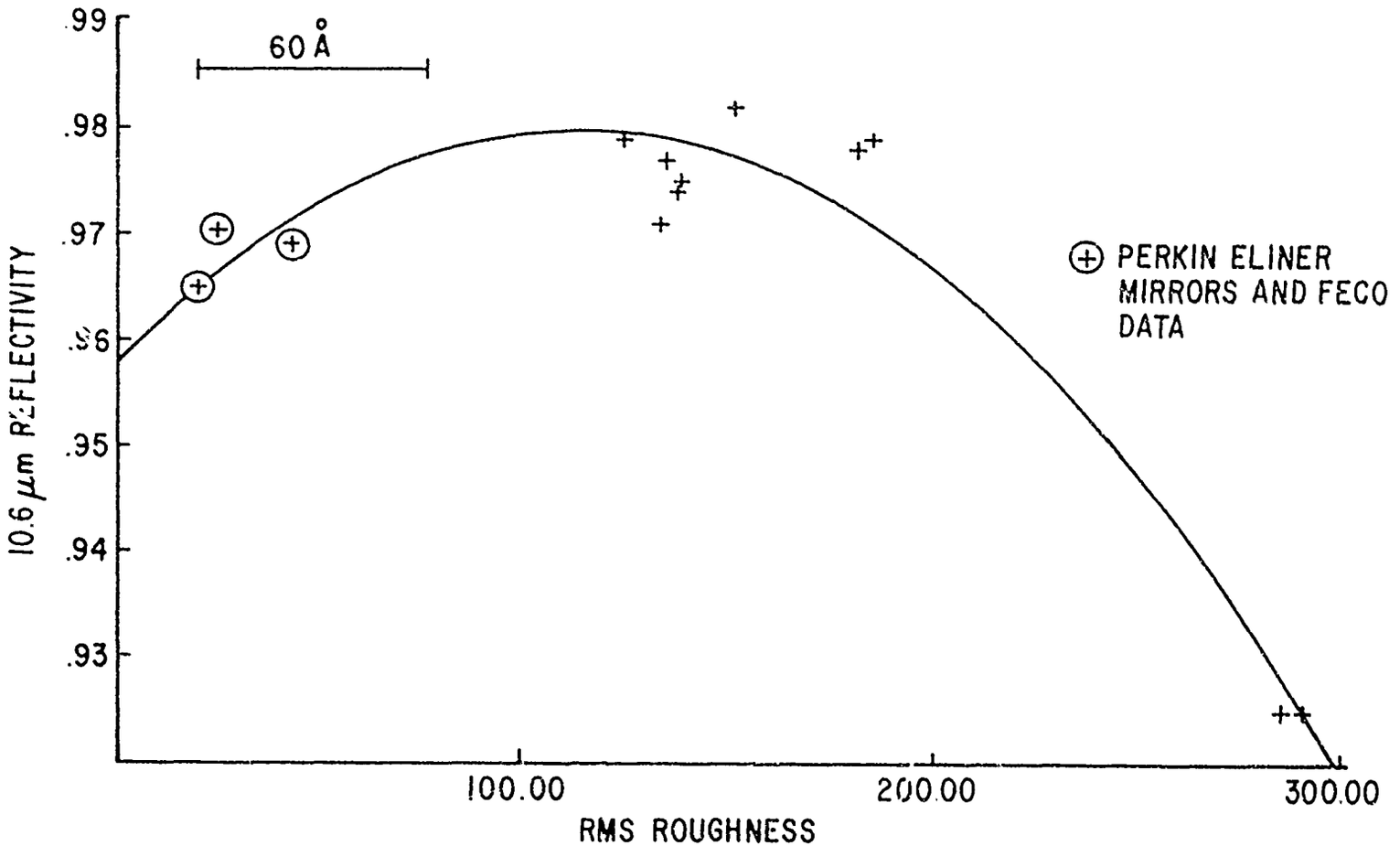
.1 mm



(58-1[illegible], M-38, [illegible] Mo[illegible], [illegible] X, center
15 Nov 73 .1 mm

(59-1), M-23, Sputtered Mo on Mo,99X
center .1 mm

MOLYBDENUM


60 Å
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10.6 μm REFLECTIVITY
100.00
200.00
300.00
RMS ROUGHNESS
⊕ PERKIN ELINER MIRRORS AND FECO DATA