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**AN INVESTIGATION OF SURFACE MODIFICATION
DUE TO THE DEPOSITION OF SILICON ONTO MOLYBDENUM**

A Thesis

Presented for the

Master of Science Degree

The University of Tennessee, Knoxville

ROBERT A. WALDROP

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ABSTRACT

This thesis reports on experiments to form a molybdenum disilicide (MoSi_2) layer on the surface of molybdenum. A 3 kW CO_2 laser was utilized to melt the surface of the molybdenum, where a powder feeder was employed to spray silicon powder into the melt pool created by the laser. A matrix of experiments was performed to find the optimal conditions for the deposition of silicon to create a MoSi_2 layer.

From these experiments it was concluded that the translation velocity of the processing laser and the amount of silicon being deposited onto the molybdenum surface are crucial parameters. The addition of silicon to the melt pool increased the cross sectional area of the fusion zone, as compared to a laser radiated surface without the addition of silicon. The addition of silicon at translation velocities of 500 and 750 (mm/min), caused a substantial increase of both the fusion zone and molybdenum-silicon areas, compared to translation velocities from 1000 to 2000 (mm/min).

The surface modification experiments produced molybdenum-silicon regions varying in percent concentrations from 1 wt. % silicon to cladding layers containing 99 wt. % silicon. Different phases of molybdenum-silicon were present including Mo_3Si , Mo_5Si_3 and a small region of MoSi_2 .

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1) translation velocity of 500 (mm/min), 2) delivered laser power of 1560 watts and 3) 4 mm beam focus. 46

CHAPTER 1

INTRODUCTION

The primary requirements of materials being used in extreme heating conditions are that they must have good oxidation resistance, high melting temperatures, high strength at elevated temperatures and high thermal conductivities. Molybdenum is one of the metals used when these high temperature requirements are present. However, the use of molybdenum for high temperature applications is limited by its rapid rate of oxidation at temperatures as low as a few hundred degrees Celsius¹. These high oxidation rates limits the uses of molybdenum as high temperature metals to only specific cases. The development of new techniques for an oxidation preventing coating for molybdenum in high temperature situations would be very useful in many different areas. Molybdenum disilicide (MoSi_2) is one such alloy that is being investigated as an oxidation prevention coating for high temperature applications, since MoSi_2 has a lower oxidation rate in elevated temperature conditions, than molybdenum alone.

This thesis is a result of an on going project supported by Arnold Engineering Development Center (AEDC) at Arnold Air Force Base, Tullahoma, TN. It is a feasibility study on extending the operational life of a molybdenum nozzle throat which experiences premature failure, as a result of its exposure to extreme temperatures and atmospheric oxygen content.

The goal of this project is to modify the surface of the molybdenum nozzle throat with silicon powder and produce an oxidation protection surface layer of molybdenum disilicide. This MoSi_2 layer can also provide an insulation barrier to help prevent thermal stresses due to sudden significant temperature changes, called thermal shock². The lower oxidation rate and thermal insulation barrier, provided by the new MoSi_2 surface layer, can possibly prevent cracking of the surface which will prolong the life of the nozzle.

This project will perform surface modification experiments by concurrently spraying the surface of the molybdenum nozzle with silicon powder and irradiating the new silicon-molybdenum surface with various CO_2 laser intensities. This procedure will form a melt pool, created by the laser, where the molybdenum melt pool and silicon can mix and form the MoSi_2 oxidation protective layer. The microstructure of the MoSi_2 surface layer and the affected regions will be examined and analyzed to determine the effectiveness of this procedure.

CHAPTER 2

BACKGROUND

This chapter provides background information on molybdenum, molybdenum disilicide, the nozzle provided by AEDC, and other surface modification techniques previously used. Data from previous test runs, that the molybdenum nozzles experiences, will also be given. Justification of using molybdenum disilicide as an oxidation protective coating will be explained.

2.1) Introduction to molybdenum

At one time, molybdenum and its alloys were regarded as a specialized aerospace material, however, today they are being employed in a wide range of conventional applications. For example they are utilized in thermal processing, electronic, nuclear, automotive, chemical glass, high temperature furnaces, and specialty alloy working industries. Molybdenum and its alloys are most often used as an alloying element in cast irons, steels, heat resistant alloys and corrosion resistant alloys to improve hardenability, toughness, and abrasion resistance. Pure molybdenum is beneficial to industry when there is a need for a material that can withstand high temperatures, severe abrasive conditions, and corrosive conditions. In the aerospace industry, molybdenum is used for high temperature structural parts including nozzles, struts, re-entry cones, heat-radiation shields, and heat engines, to name only a few.

Molybdenum and its alloys are particularly advantageous for airframes because of their retention of high stiffness, good creep strength, and high recrystallization temperature, after thermal cycling¹.

Desirable characteristics of molybdenum include, that it has a relatively low density of 10.22 g/cc (compared to tantalum and tungsten), it has a high modulus of elasticity of 46×10^6 psi, excellent retention of strength at high temperatures, and that it is relatively abundant^{3,1}. Other properties of molybdenum are, that it has a high melting temperature of 2610 °C, it has body centered cubic (BCC) crystal structure, and that it begins to oxidize dramatically at temperatures above 500 °C⁴. These properties of molybdenum classifies it as a refractory metal. These and more characteristics have made molybdenum a high temperature application material. A more detailed list of physical properties for molybdenum is given in Appendix A.

Since molybdenum begins to oxidize at a low temperature, in an oxidizing atmosphere, it is a major disadvantage for usage in a high temperature regime (> 500 °C). In this environment the formation of the oxide, molybdenum trioxide (MoO_3) occurs and can lead to extreme oxidation. Due to intense volatilization by the oxidation of MoO_3 there is a sharp reduction of mass of the molybdenum object^{5,6}. This oxidation can cause total failure in the molybdenum material.

2.2) Benefits of molybdenum disilicide

Due to significant improvements in high performance aerospace systems, such as ground test facilities that simulate high speed flight, a renewed interest in

manufacturing structural materials capable of operating at high temperatures without oxidizing has emerged. There have been many ideas to increase the oxidation resistance of the refractory metals such as molybdenum, columbium and titanium. One approach is to develop refractory inorganic coatings to protect these alloys from oxidation. Another alternative is to develop a slow oxidizing, high temperature coating. This coating would need to be a thin, high temperature, low oxidation coating on the surface of the base material that possesses these and other characteristics desired. This coating should also not affect the desired characteristics of the base material being considered¹. This approach is the basic concept behind this project.

Molybdenum disilicide is one material that is presently undergoing research as an oxidation prevention coating, since MoSi_2 is considered a self healing and protective coating in oxidizing environments. One advantage of MoSi_2 is that it provides an excellent protective oxidation layer. This layer protects the base material because it does not begin to oxidize until 1800 °C. This oxidation temperature is much higher than the 500 °C temperature at which molybdenum begins to oxidize¹. Another advantage of a MoSi_2 coating is that it acquires a protective, self healing film of silicon dioxide (SiO_2) in oxidizing atmospheres. The self healing process is achieved due to the formation of SiO_2 in cracks, which essentially seals the cracks. This self healing occurs from the growth of pure SiO_2 protective scales, from the MoSi_2 layer. The SiO_2 protective scales form when the oxides of molybdenum evaporate during the oxidation process. Subsequent oxidation is then regulated by molecular diffusion of oxygen through the SiO_2 layer⁷. A further advantage of MoSi_2 is that it possesses a low

coefficient of thermal expansion, which reduces thermal stresses and thus improving thermal shock resistance¹. A more detailed list of molybdenum disilicide's physical properties are given in Appendix C.

There exists a number of silicide compounds that have good oxidation resistance characteristics, similar to molybdenum disilicide. However, only a limited number of the silicides have been considered for mechanical property application. This could be due to poor thermal stabilities of these silicides. For example, the silicon-base ceramics such as silicon carbide (SiC) and silicon nitride (Si₃N₄) have excellent oxidation resistance, high melting points and good strength properties. However, they lack high temperature ductility, are difficult to alloy with other elements, and are difficult to machine. Further investigation shows that the MoSi₂ materials can be more reliable and less expensive to manufacture than the silicon-base ceramic materials².

2.3) Limitations of molybdenum disilicide

It has already been determined that MoSi₂ is beneficial in the uses as an oxidation prevention coating, for high temperature situations. However, there are drawbacks that have to be considered when using this material as an protective coating. As stated earlier, in an oxidizing atmosphere the MoSi₂ coating acquires a protective SiO₂ coating. This coating is not the only phase formed. Pentamolybdenum trisilicide (Mo₅Si₃) is also formed, which is not self healing. The Mo₅Si₃ phase is formed due to oxidation of the MoSi₂ coating. This oxidation depletes the MoSi₂ coating of silicon and forms the lower Mo₅Si₃ silicide phase which does not form protective silica scales

and thus oxidizes rapidly^{8,9}. To prolong the life of the MoSi₂ protective coating, thereby prolonging the life of the molybdenum base material, the loss of silicon must be prevented in the MoSi₂ coating⁹.

Another disadvantage is that the thermal-mechanical limitations of MoSi₂ coatings can cause thermal stresses in the coating. Stresses created from the variances in the coefficient of thermal expansion of the MoSi₂ and the molybdenum base material can produce microcracks in the MoSi₂ coating layer. The microcracks can form, if these stresses from the variances in the coefficient of thermal expansion exceeds the strength of the MoSi₂. These cracks can possibly cause chipping of the protective silica composition which can result in silicon depletion from the MoSi₂ layer and lead to rapid coating degradation⁸.

Another property of MoSi₂ that is a drawback is its brittle-to-ductile transition of 900 - 1000 °C. This lack of ductility at low temperatures have caused difficulties in the processing of fabricating specimens, which have led to its restrictive studies of its behavior².

2.4) History of the molybdenum nozzle

The molybdenum nozzle provided by AEDC is from the Free Piston Impulse Tunnel, in Tullahoma, TN. This facility is a high performance, free piston shock tunnel, used to perform hypersonic model tests. A cross section diagram of the nozzle is given in Figure 2.1. The tunnel generates extremely high shock tunnel reservoir conditions in these hypersonic model tests. The ultimate test conditions desired reach

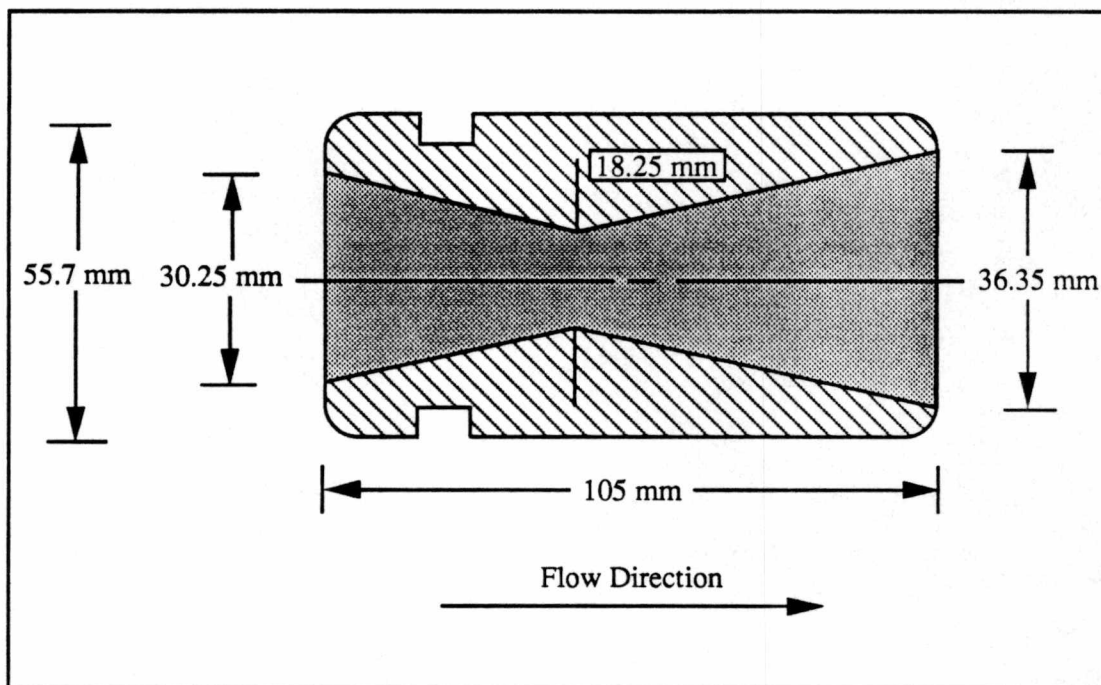


Figure 2.1 A cross section diagram of the molybdenum exhaust nozzle, provided by Arnold Engineering Development Center (NTS).

reservoir pressures of 10,000 atm and reservoir temperatures of 10,000 K, with duration times of 3 - 10 ms. These test conditions result in intense heating of the nozzle throat with a heat transfer rate of approximately 750,000 BTU/ft²-sec. This induces melting, vaporization or ablation, and oxidation of the throat materials. These problems reduce the duration times and restrict the test conditions¹⁰.

Information provided from previous test runs from the Impulse Facility at AEDC, shows that the nozzles are subjected to stagnation pressures from the range of 3250 (psi) to 17,000 (psi), with stagnation temperatures of 5640 K to 8430 K, and enthalpy rates from 4080 (BTU/lb) to 6940 (BTU/lb). This information also states that under these conditions, the nozzle experiences cracks about every third test run. These cracks can result in the complete failure of the nozzle¹¹.

2.5) Other methods of surface alloying

The desire to develop new high temperature materials has sparked many ideas to prolong the life of fast oxidation materials. One such method is plasma spraying of base materials with silicides, to be used as an oxidation protective coating. Previously, low pressure plasma-spraying methods (LPPS) has been used to coat base materials with silicides, for an oxidation protective coating⁹. The LPPS process consists of heating a gas with a high-current arc, creating plasma. The required powder is then injected into the plasma, creating a plasma enriched with the desired powder concentration. The plasma with the melted powder vapor, is then directed onto the surface of the base material, where it is applied as a coating. In particular, MoSi₂ has

been injected into plasma, where it was used to coat a molybdenum sample. This method has proven successful in creating a MoSi_2 coating. However in the process, transverse cracks in the MoSi_2 layer was created, and also layer separation was observed. These results are believed to be due to stress resulting from different linear expansivities of MoSi_2 and molybdenum. These cracks are detrimental to the oxidation protective coating⁹.

Other surface treatment methods utilize high power lasers for alloying of materials. One of these methods is to initiate a phase transformation process by employing a laser to irradiate the material surface. Grain refinement, rapid solidification, and amorphous structures is beneficial from the molten phase created by the laser, in this treatment process. The phase transformation process can be called a surface heat treatment process whereas conventional processing is limited to martensitic transformation only¹².

Another method is compositional transformation which has been around for over 20 years. This method employs cladding for improving surface properties. Cladding consists of attaching a material of different chemical composition to a base material, to improve its surface properties. In this method, to create the new composition, a laser heats the base material, where the desired cladding material is applied to the surface. Powders have been utilized as a cladding material, but wire, rod, and strip can also be used, depending on the properties desired. Stainless steel has successfully been employed as a cladding to rebuild some parts, to increase their corrosion resistance. This process can keep the overall cost of the part down¹².

CHAPTER 3

EXPERIMENTAL PROCEDURE

In this chapter the experiment parameters for the single track, multiple track, single and multiple track with silicon powder and single track silicon cladding with remelting track will be described. The equipment used will also be described for this study of surface modification from the deposition of silicon onto molybdenum.

3.1) Single track and multiple track procedures

The objective of these measurements was to determine the proper power, translation velocities and overlap widths to be applied throughout the experiments. This was accomplished by irradiating the surface of a molybdenum sample with various intensities of an incident continuous wave laser pulse with different translation velocities. The results were compared with results of the experiments with the addition of silicon, to investigate the metallurgical changes due to the addition of silicon.

A Rofin Sinar 3 kW CO₂ laser was operated in Pulse Width Modulation mode (PWM) with a beam focused 2 mm above the molybdenum surface, which was used to irradiate the molybdenum surface, under investigation. Helium, with a flow rate of 30 (liters/min), was used as the coaxial shield gas in the laser's delivering nozzle. The laser is equipped with a computer numeric controlled (CNC) work table that secures

the material being treated and is capable of moving in X and Y directions in micron step increments.

An IMACON 790 high speed camera photographed the surface area, taking nine frames on one photograph during a single surface modification run. The time interval between each frame was 10^{-4} frames/second. The nine frames on each photograph were taken sequentially over the span of 1 ms.

An Argon Ion laser beam provided the proper lighting conditions for the IMACON high speed camera. This required that the Argon Ion laser's operating current to be set at 200 amps and a neutral density filter be placed between the reflected beam and the high speed camera, to help control the beams intensity. A diagram of the experiment setup is shown in Figure 3.1.

A pulse generator and a triggering circuit generated the required voltage and time delay to the IMACON high speed camera. The triggering circuit output voltage was 10 volts, which was sufficient to trigger the high speed camera. The triggering circuit was set for a delay of 0.2 seconds after the CO₂ laser triggered. This delay made certain that the high speed photographs would be taken after the surface modification runs were in progress. An oscilloscope was in series with the triggering circuit to verify camera triggering. A diagram of the triggering sequence is shown in Figure 3.2.

For the single track experiments, the surface of the molybdenum was subjected to various laser output intensities at four different translation velocities. A table of these powers and translation velocities is shown in Table 3.1. The delivered powers

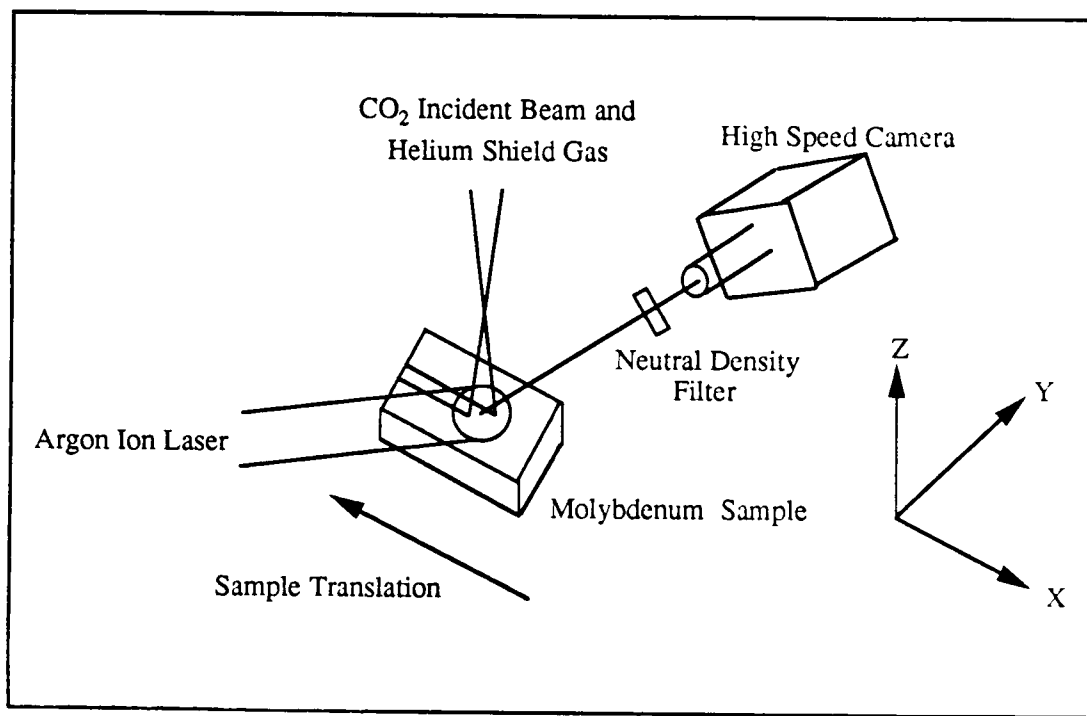


Figure 3.1 Schematic diagram of the experimental setup for single and multiple track runs.

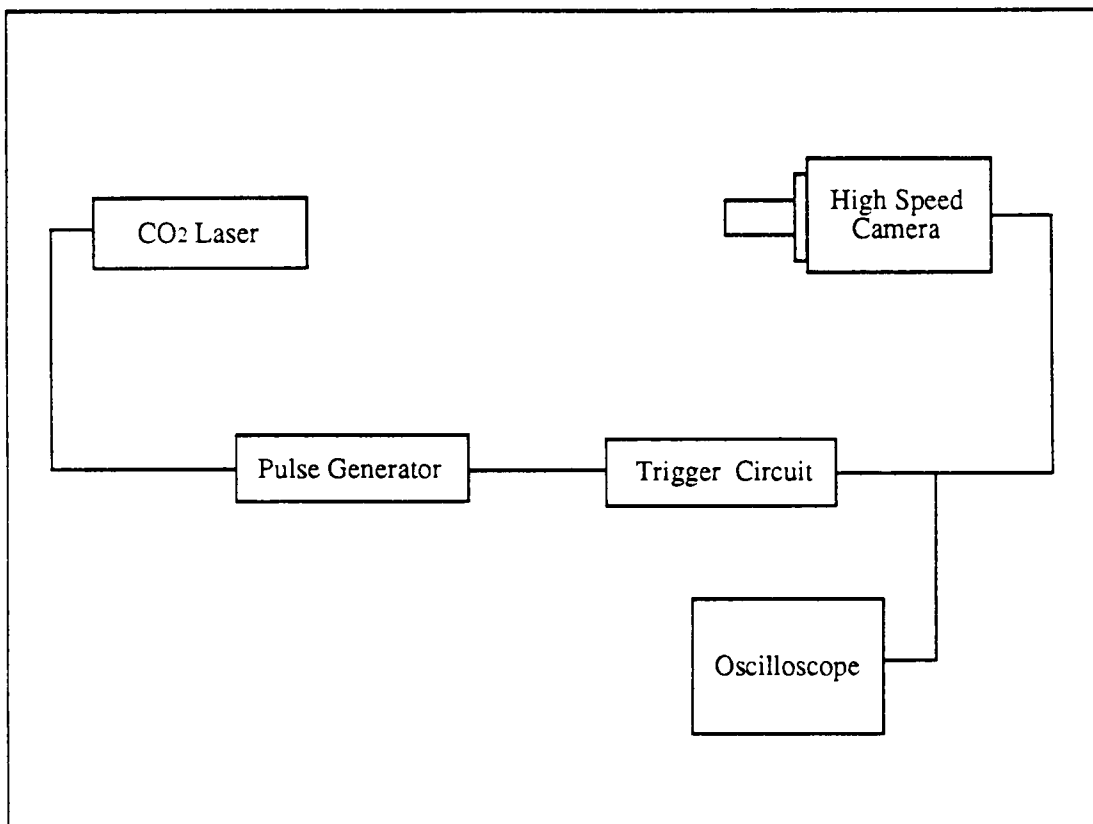


Figure 3.2 Triggering block diagram for single track and multiple track experiments.

Table 3.1 CO₂ delivered laser powers with a beam focus of 2 mm above the surface and translation velocities for single track without silicon experiments.

Laser Power (W)	Translation Velocities (mm/min)			
2552	500	750	1250	1500
2350	500	750	1250	1500
2244	500	750	1250	1500
2154	500	750	1250	1500
2050	500	750	1250	1500
1700	500	750	1250	1500
1500	500	750	1250	1500
1000	500	750	1250	1500

were measured by using a Scientech 390 Calorimeter. The consistency of the delivered output powers were verified by measuring the powers before and after the experiments. The accuracy of the calorimeter's measurements was confirmed by using a power stick to measure the laser's delivered output powers. To ensure the same thermal operating conditions, the temperature of the molybdenum samples was monitored in every experiment with a thermocouple before and after each experimental run.

The equipment set up for the multiple track experiments was the same as in the single track measurements. These multiple track experiments were conducted in two series. One series applied an overlap of 50 % of the original single track width and the other series applied a 75 % overlap. Both of the overlap cases operated with a CO₂ laser delivered power of 2600 watts and three translation velocities. Table 3.2 lists the multiple track experiment parameters.

Table 3.2 Operating parameters for 5 multiple tracks without silicon runs.

CO ₂ Laser Delivered Power	2600 watts
Translation Velocities	750, 1250, 1500 (mm/min)
Overlap Specifications	50 % overlap, displacement width of 280 μ m 75 % overlap, displacement width of 140 μ m
Beam Focus	2 mm above molybdenum surface
laser Pulse Type	Continuous Wave in PWM mode

3.2) Single track and multiple track with the deposition of silicon powder procedures

This series of experiments investigated the metallurgical changes of the molybdenum due to the addition of silicon powder. Single track runs with the addition of silicon were performed to determine the correct overlap widths to be used in the multiple track procedure. A specially designed powder feeder was employed to spray -325 mesh, 99 % pure silicon onto the surface of the molybdenum sample at an angle of approximately 50 degrees concurrently with the operation of the CO₂ laser. During each experimental run a Nikon camera photographed the modified areas, using a 35 mm lens to magnify the area of interaction. A diagram of the set up for the experiment is shown in Figure 3.3.

The single track with silicon powder experiments utilized a measured delivered CO₂ laser power of 2600 watts with four translation velocities. Since the powder

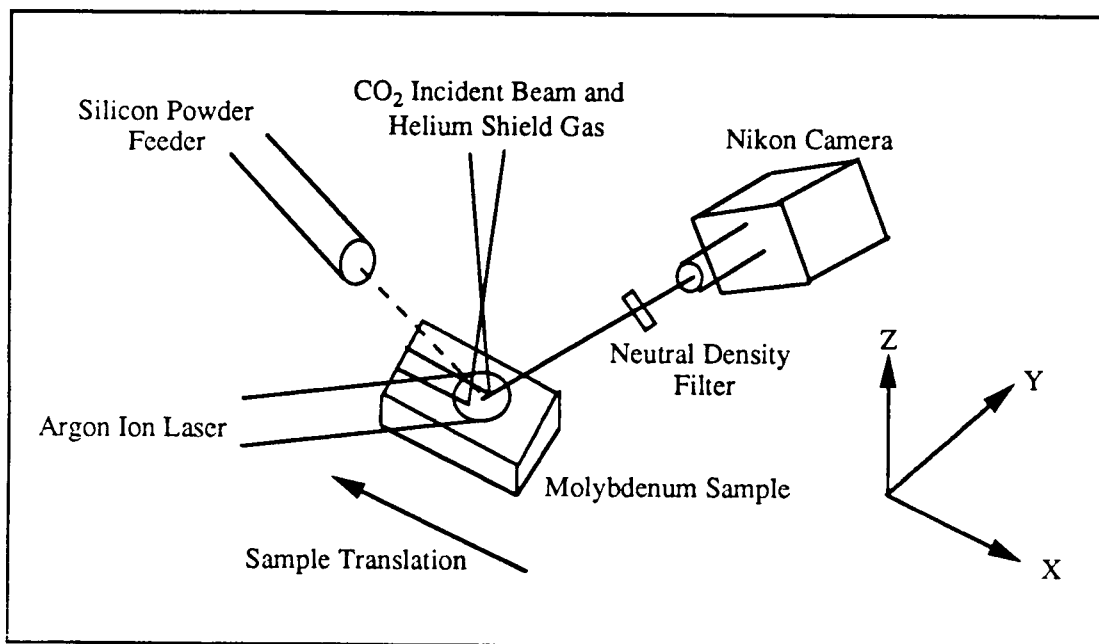


Figure 3.3 Schematic diagram of the experimental setup for multiple track runs with the addition of silicon powder.

feeder output rate was constant, four stand off distances (the end of the powder feeder nozzle to the molybdenum surface) were used to regulate the amount of silicon being sprayed onto the melted surface region of the molybdenum sample. These parameters are located in Table 3.3. Analysis of the results from these experiments can provide information concerning the relation between the powder feeder stand off distance and the silicon concentration in the molybdenum-silicon regions. After each experimental run with silicon, the surface of the molybdenum sample was cleaned with acetone.

In the multiple track with silicon powder experiments, one overlap of 50 % of the molybdenum-silicon layer created from the previous experiment, was determined and used. An illustration on determining this overlap is given in Figure 3.4. This overlap ensured a concentrated, consistent layer of silicon being introduced into the melt pool of the molybdenum sample. A CO₂ laser delivered power of 2600 watt

Table 3.3 Operating parameters for single track with silicon powder runs.

CO ₂ Laser Delivered Power	2600 watts
Silicon Powder Feeder Rate	2.75 (g/min)
Stand Off Distance	3 mm, 2.2 cm, 3.2 cm, 4.2 cm
Translation Velocities	2000, 1500, 1250, 1000, 750, 500 (mm/min)
Beam Focus	2 mm above the molybdenum surface
Laser Pulse Type	Continuous Wave in PWM mode

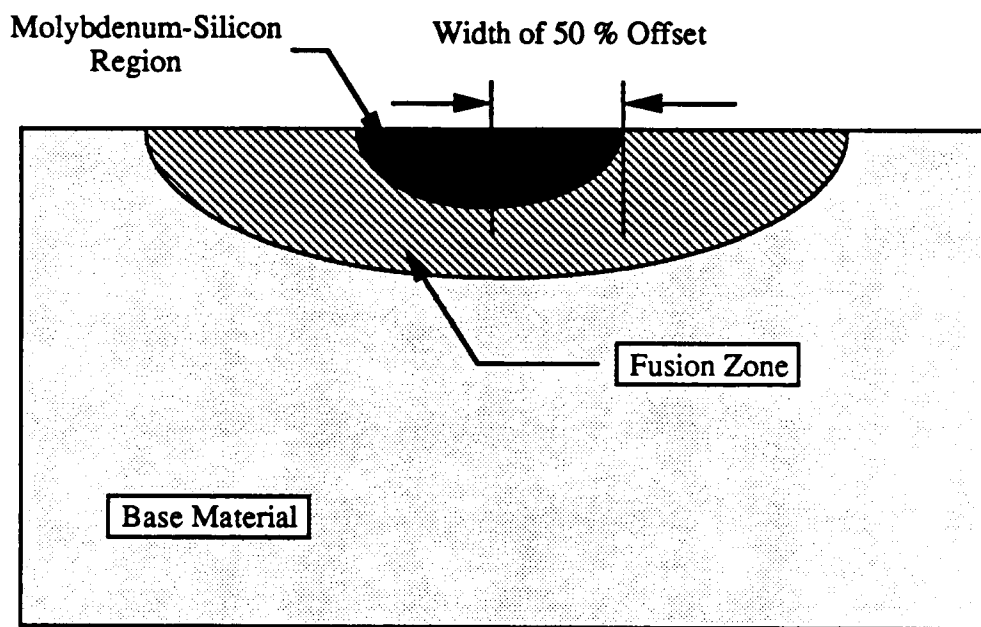


Figure 3.4 Illustration showing the determination of the 50 % overlap width for the multiple track with silicon experiments.

with a powder feeder rate of 2.75 (g/min) and a stand off distance of 2.2 cm was used in this procedure. From the results of the single track with silicon, two translation velocities, 1000 and 1500 (mm/min), were selected for further study. The parameters of this experiment are shown in Table 3.4.

3.3) Single track silicon cladding with remelting track procedure

This series of experiments consisted of irradiating the molybdenum surface with a delivered power of 2600 watts, while spraying the surface with silicon powder at a stand off distance of 3 mm. At this distance a cladding layer can form on the surface of the molybdenum sample. This cladding layer was then subjected to a remelting pass with a beam focussed 4 mm above the molybdenum sample surface. Remelting passes

Table 3.4 Operating parameters for 5 multiple tracks with silicon powder runs.

CO ₂ laser Delivered Power	2600 watts
Silicon Powder Feeder Rate	2.75 (g/min)
Stand Off Distance	2.2 cm
Translation Velocities	1000, 1500 (mm/min)
Overlap Specifications	50 % overlap, displacement width: 210 μ m
Beam Focus	2 mm above molybdenum surface
Laser Pulse Type	Continuous Wave in PWM mode

were conducted with two different laser intensities and translation velocities. A matrix of these parameters is located in Table 3.5

3.4) Metallurgical analysis

Metallurgical analysis was conducted to examine the metallurgical changes that occurred in the molybdenum sample due to the laser radiation, the silicon powder addition and the remelting tracks. Overview photographs of the molybdenum samples were taken using a Nikon SMZ-U stereo microscope. Each of the samples were cross sectioned with a high speed diamond wheel saw and then mounted in an epoxy mold. A diagram of the cross section locations is in Figure 3.5. The mounted samples were polished on metallographic polishing wheels with increasing grit size starting from grade 60 to 600. The samples were polished on metallographic wheels using a polishing cloth with alumina suspension as an abrasive. The polished samples were then etched with Maurikamis solution to reveal the microstructure of the fusion zone, molybdenum-silicon region and base material.

A Buehler Micromet II Microhardness Tester with microscope which has a graduated X-Y translation stage, was used to measure the width and depth of the affected regions. An investigation for cracks in the regions was performed and the size of existing cracks were measured, if present.

Photographs of the cross sectional microstructure areas were taken with an Olympus Inverted Metallurgical Microscope. This microscope has a 35 mm camera attachment and is capable of photographing with various magnifications. A Cameca

Table 3.5 Operating parameters for single track silicon cladding with remelting track procedure including an initial track_(IT) laser power of 2600 watts, remelting tracks_(RT), and a powder feeder stand off distance of 3 mm.

Translation Velocity _{IT} (mm/min)	Translation Velocity _{RT} (mm/min)	Laser Power _{RT} (watts)	Beam Focus _{IT} above surface	Beam Focus _{RT} above surface
1000	500	1040	2 mm	4 mm
1500				
2000				
1000	500	1560	2 mm	4 mm
1500				
2000				
1000	1000	1040	2 mm	4 mm
1500				
2000				
1000	1000	1560	2 mm	4 mm
1500				
2000				

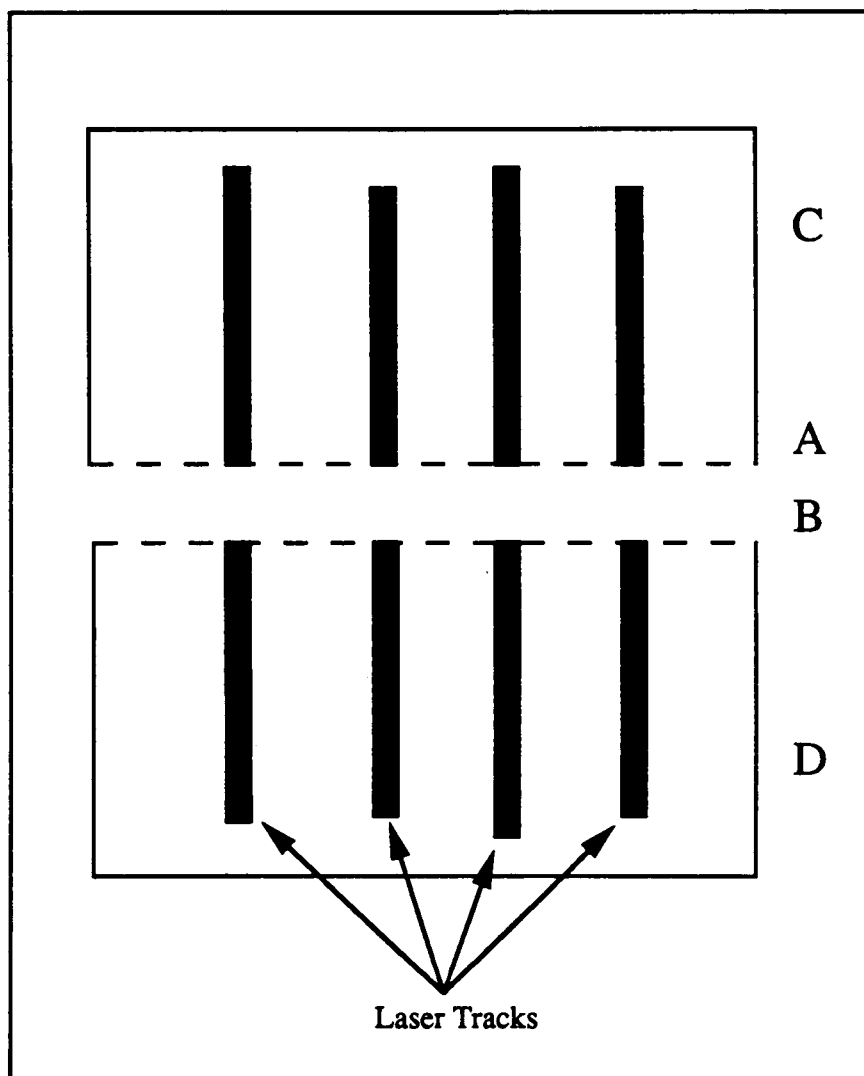


Figure 3.5 Cross section planes for the molybdenum sample.

scanning electron microprobe analyzer from NASA's Metallurgical and Processing Laboratory in Huntsville, AL was utilized for high resolution scanning electron microscopy, elemental analysis and photographing the microstructure.

CHAPTER 4

RESULTS AND DISCUSSIONS

This chapter gives the results of all the experiments conducted. Photographs of the molybdenum samples from the experiments will show the melted area called the fusion zone, the base material, and the molybdenum-silicon layers. Analysis and photographs of the silicon content of the molybdenum-silicon layers, using a scanning electron microprobe will be provided. Results of the experiments will be discussed.

4.1) Results from the single track and multiple track experiments

The data obtained from the single track experiments is shown in the graph in Figure 4.1, which shows that the depths of the fusion zones decreased as the translation velocity increased. The slight variations in the depths of penetration can be attributed to inadequate laser-surface interaction.

Results of the single track experiments indicate that the fusion zones formed by the laser powers below 2500 watts were too small to form the melt pool that is needed for the addition of silicon experiments. Without the proper melt pool the silicon provided will not be able to mix with the molten molybdenum to form the desired MoSi_2 layer. Throughout the rest of the experiments the delivered laser powers, were kept at approximately 2500 watts or above.

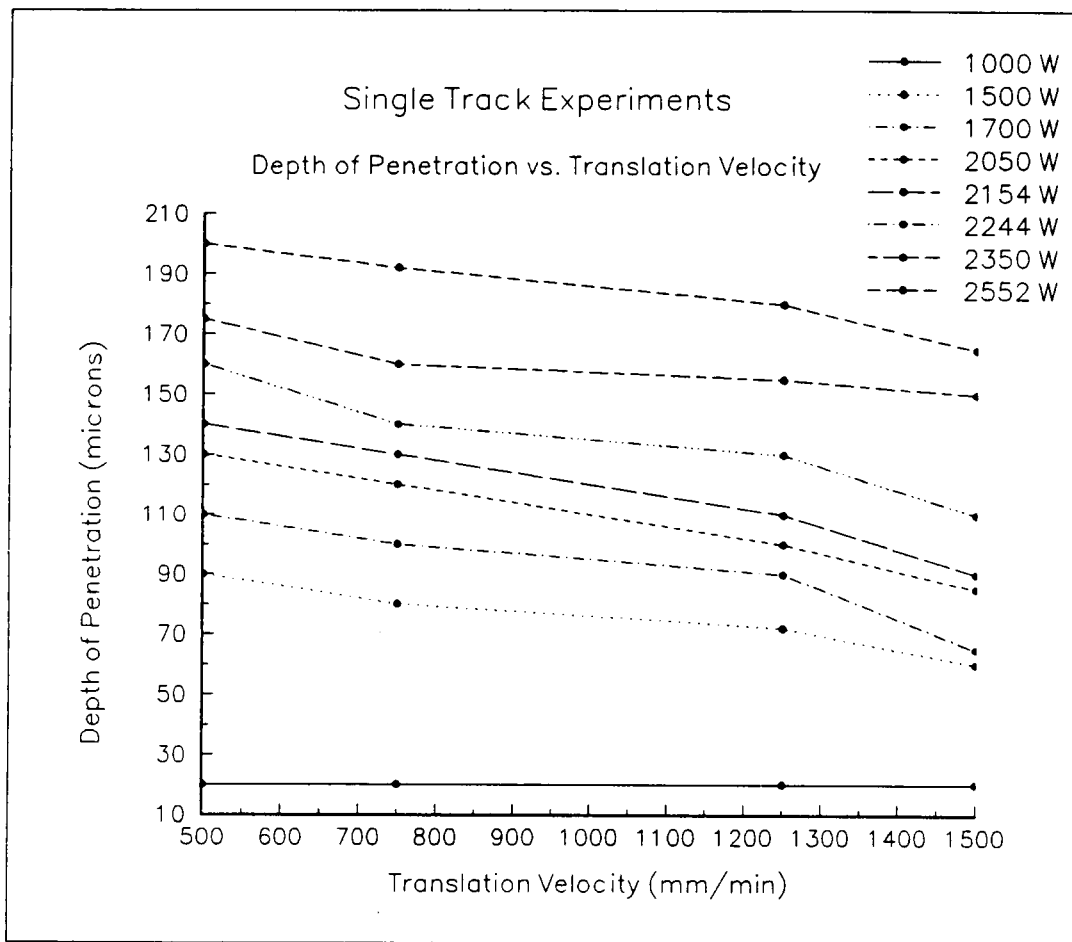


Figure 4.1 Graph of Depth of Penetration vs. Translation Velocity for the single track without silicon experiments, using a range of laser delivered powers.

For these conditions, cracks were not apparent in any of the samples for the powers used. This indicated good interaction of the laser radiation on the molybdenum sample, without creating harmful stresses in its microstructure. A photograph of an untreated, polished and etched cross section of the molybdenum nozzle sample is shown in Figure 4.2 (a). This photograph shows the elongated grains that develop from a typical rolling procedure performed in the processing of the material. Figure 4.2 (b) is a photograph of the desired fusion zone of a polished and etched molybdenum sample which was irradiated by the laser with a delivered energy of 2600 watts, with a translation velocity of 1250 (mm/min), and at a focal point of 2 mm above the surface. For this sample, the depth and width of the fusion zone is approximately 210 μm and 590 μm , respectively. A photograph showing a fusion zone, with a molybdenum-silicon region, is given in Figure 4.3. From the results of the single track experiments, the distances for the 50 % and 75 % overlaps were determined for use in the next procedure, the multiple track without silicon experiments.

From the results of the multiple track without silicon experiments it was determined that there is little variation in the depth of the fusion zones between the 50 % and 75 % overlaps. It also was determined that the widths of the fusion zones decrease as the translation velocity increases, which was expected. In these cases, cracks were not apparent indicating that the molybdenum sample can withstand thermal shock and other stress due to the intense heat and shock of the laser energy without cracking the material. High speed photography by the IMACON camera shows nothing conclusive about the laser-surface interaction of the molybdenum sample.

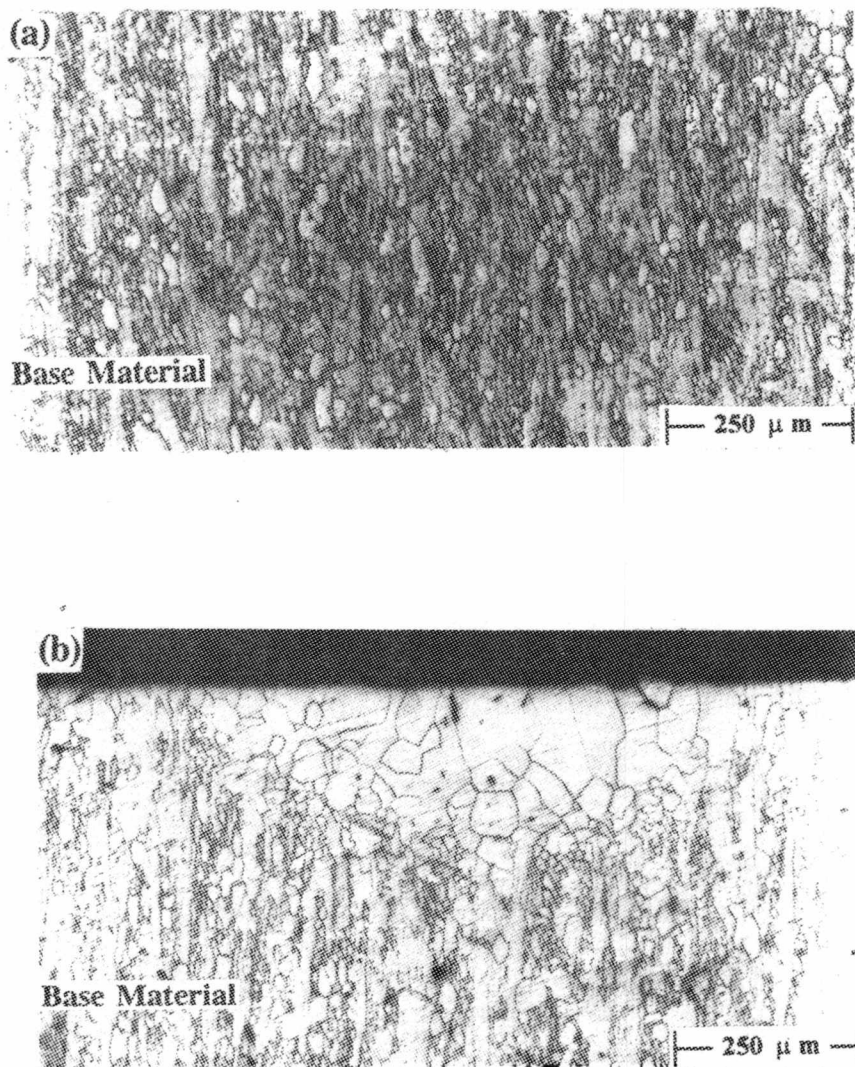


Figure 4.2 Optical photographs of a molybdenum sample with (a) an untreated surface and (b) an ideal fusion zone formed with a delivered power of laser 2600 watts and with a translation velocity of 1250 (mm/min).

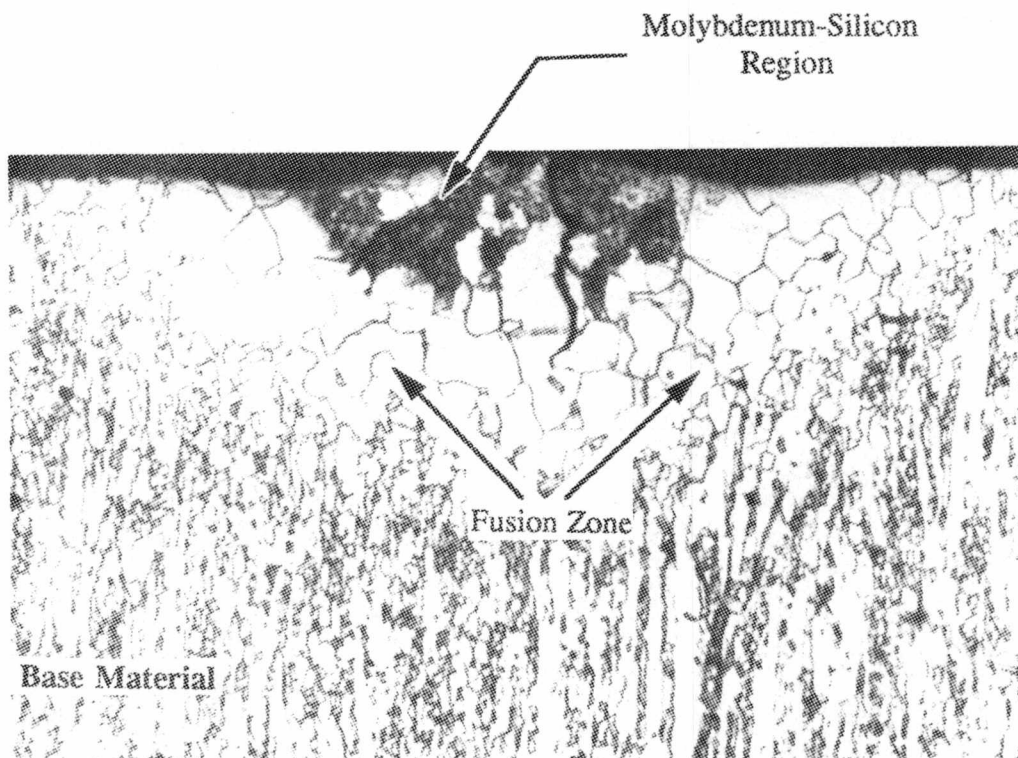


Figure 4.3 Optical photograph illustrating the base material, fusion zone and molybdenum-silicon layer.

4.2) Results from the single track and multiple track with silicon experiments

The single track with silicon experiments using different stand off distances were performed to determine how much silicon should be sprayed into the melt pool. The stand off distance in conjunction with the translation velocity essentially regulates the amount of silicon being projected into the melt pool. Since the required atomic percent concentration to form a MoSi_2 compound is 37 % Mo and 63 % Si, the amount of silicon being sprayed into the melt pool is very important. This will require 66.75 wt. % Mo and 33.25 wt. % Si to form MoSi_2 ¹³. These compositional percentages are the percentages that these experiments are seeking.

Stand off distances of 3 mm, 2.2 cm, 3.2 cm, and 4.2 cm, with translation velocities between 500 and 2000 (mm/min) and with a laser delivered power of 2600 watts were used in forming the molybdenum-silicon regions. The depths and widths of the silicon enriched regions were determined from the differences of the etchant characteristics for the molybdenum base material and the molybdenum-silicon regions. At a stand off distance of 3 mm and translation velocities of 750, 1250, and 1500 (mm/min), the silicon powder overwhelmed the melt pool created by the laser. This resulted in the formation of a cladding layer of silicon containing cracks, on the molybdenum surface. A photograph of a sample cross section with three single tracks with silicon at a translation velocity of 1500 (mm/min), resulting in a cladding depth of 80 μm is given in Figure 4.4 (a). Scanning electron analysis by the microprobe determined that this layer consisted of almost 99 wt. % silicon. Figure 4.4 (b) is a photograph of two single tracks with silicon formed with a translation velocity of

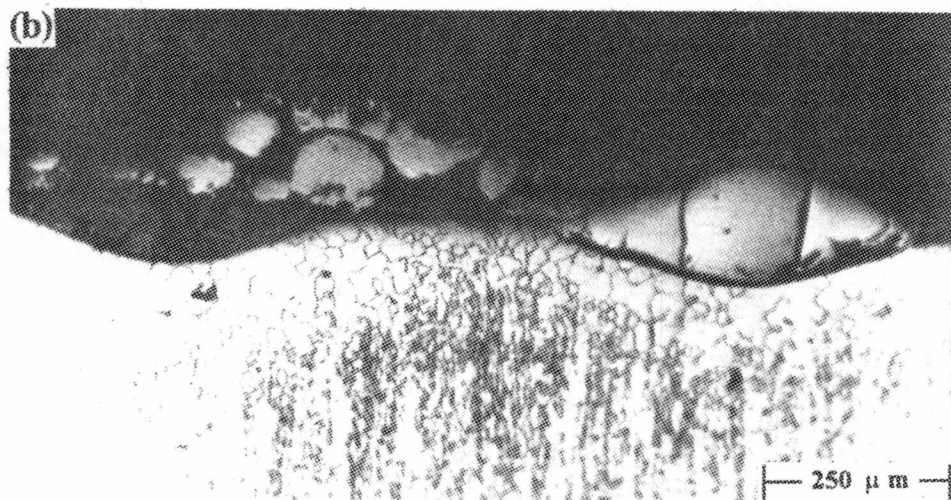
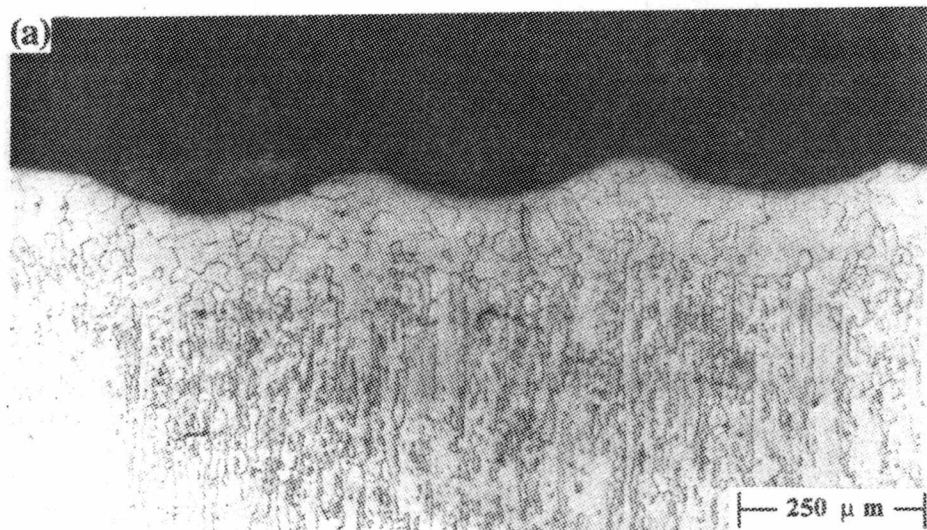


Figure 4.4 Optical photographs of single tracks with silicon at a stand off distance of 3 mm with translation velocities of (a) 1500 (mm/min) and (b) 1250 (mm/min).

1250 (mm/min), resulting in a cladding layer depth of 160 μm . Microprobe analysis shows that this mixture is mainly 99 wt. % silicon. However, in small sections located around the boundary between the silicon cladding layer and the base material, there exists different concentrations of molybdenum-silicon. The sections include concentrations indicating the presence of molybdenum-silicon phases of Mo_3Si , Mo_5Si_3 , and a small layer of MoSi_2 . These phases can be identified on the molybdenum-silicon phase diagram¹³ located in Figure 4.5. Back scattered scanning electron images (obtained by the scanning electron microprobe) identify the MoSi_2 region in Figures 4.6 (a) and (b).

With a translation velocity of 750 (mm/min) and a 3 mm powder feeder stand off distance, there was a dramatic increase of the cross sectional area of the fusion zone and the cladding layer sizes, compared to the samples with translation velocities of 1250 and 1500 (mm/min). A photograph of the cladding layer with a maximum depth of 1150 μm is shown in Figure 4.7. Microprobe analysis of this cladding layer determined that various regions of the cladding layer contained different weight percent concentrations of molybdenum and silicon ranging from 99 % Mo and 1 % Si to 90 % Mo and 10 % Si. However, in every case the cladding layers contained cracks throughout the regions. Table 4.1 gives the depths of these fusion zones and the cladding layers for the translation velocities of 750, 1250, 1500 (mm/min).

Table 4.2 consists of the results of the experiments performed with a delivered laser power of 2600 watts, a stand off distance of 2.2 cm and translation velocities of 500, 1000, 1500, and 2000 (mm/min). From the results of these experiments, it was

Mo-Si

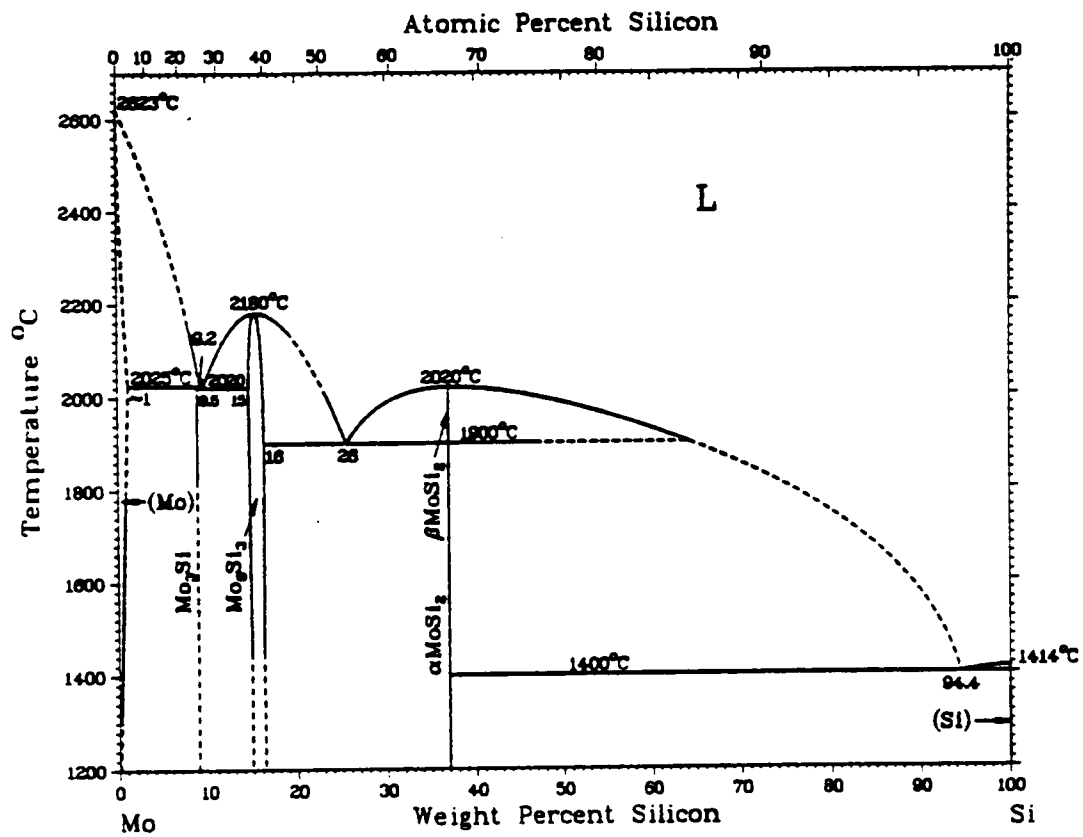


Figure 4.5 Molybdenum-Silicon phase diagram¹³.

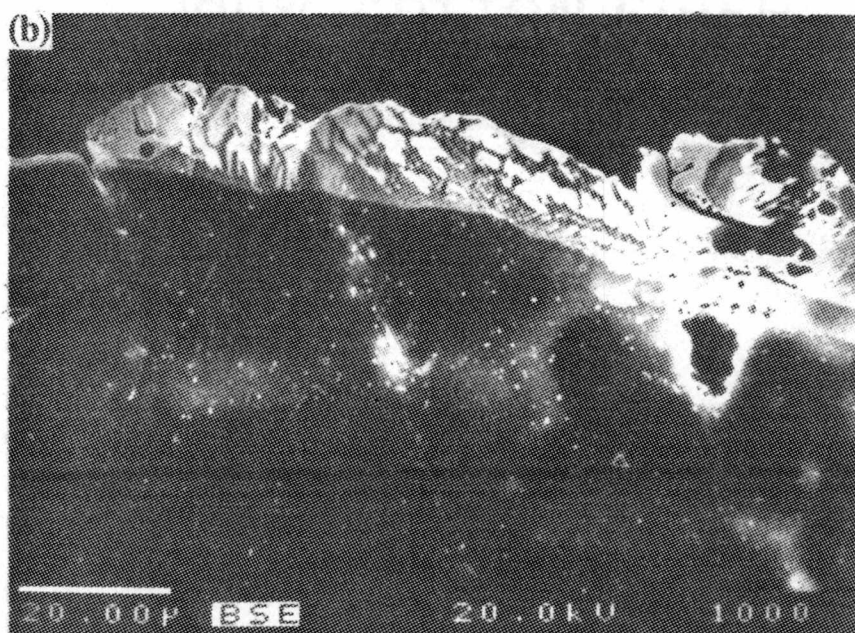
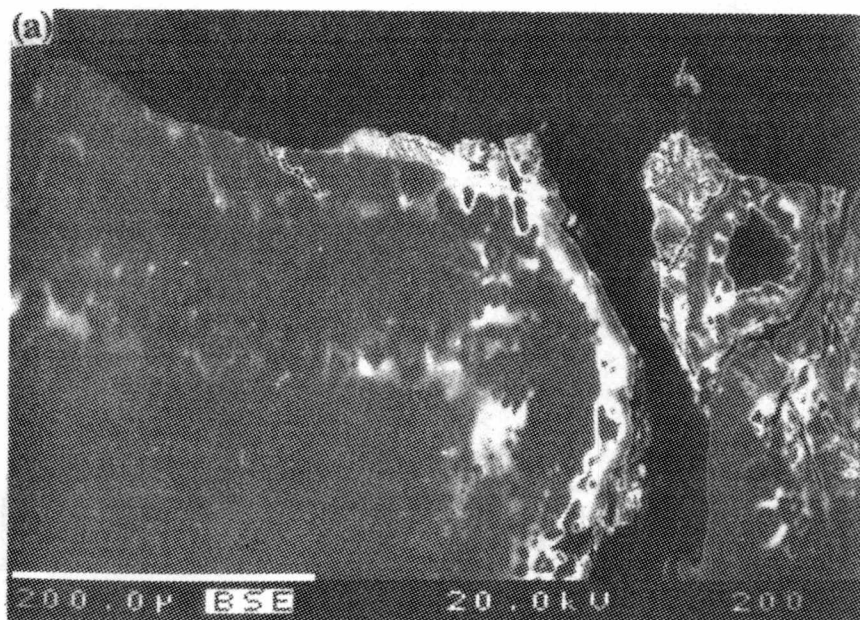


Figure 4.6 Back scattered scanning electron image showing the MoSi_2 region with magnification of (a) 200X and (b) 1000X.

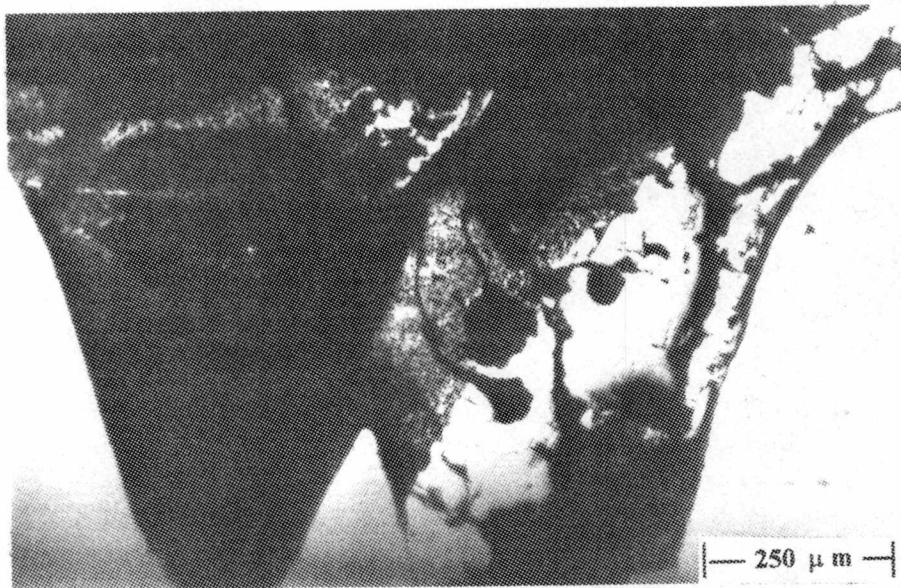


Figure 4.7 Optical photograph of two single tracks with silicon at a stand off distance of 3 mm and a translation velocity of 750 (mm/min).

Table 4.1 Table giving the fusion zone and the cladding layer depths, with a powder feeder stand off distance of 3 mm.

Laser Power (watts)	Translation Velocity (mm/min)	Fusion Zone Depth (μm)	Cladding Layer Depth (μm)
2600	750	1250	1150
	1250	270	160
	1500	160	80

Table 4.2 Table giving the fusion zone and molybdenum-silicon depths with a powder feeder stand off distance of 2.2 cm.

Laser Power (watts)	Translation Velocity (mm/min)	Fusion Zone Depth (μm)	Mo-Si Layer Depth (μm)
2600	500	950	800
	1000	300	160
	1500	250	140
	2000	220	110

determined that with the translation velocity of 500 (mm/min), there was a dramatic increase of the cross sectional area of the fusion zone and the molybdenum-silicon concentrated region, compared to the regions with the translation velocities of 1000, 1500 and 2000 (mm/min). A graph of the results from the experiments of the four translation velocities is shown in Figure 4.8. Microprobe analysis of these regions, for all four samples, determined that the concentrations of the molybdenum-silicon mixtures are less than 2 % silicon. In photographs given in Figure 4.9 (a) and (b), the sizes of the fusion zones for the case of translation velocities of 500 and 1000 (mm/min), can be compared. In these cases, transverse cracks appeared throughout the molybdenum-silicon layers, as well.

The increased fusion zones with the lower translation velocities, 500 and 750 (mm/min), can be attributed to the higher absorptivity and the thermal conduction characteristics of the silicon, compared to molybdenum. These slower translation velocities allow the silicon to absorb the heat energy of the CO₂ laser and conduct it to the molybdenum base material. This, in turn forms a larger fusion zone and molybdenum-silicon zone, compared to the faster translation velocities.

With stand off distances of 3.2 cm and 4.2 cm, microprobe analysis determined that the concentration weight percentage silicon was less than 1 %. These regions also contained small cracks in the fusion zones.

One objective of surface modification is to have the smallest fusion zones possible for the modified regions, since the fusion zones contain larger grains, which are weaker and have higher stresses than an unaffected region. For the case of

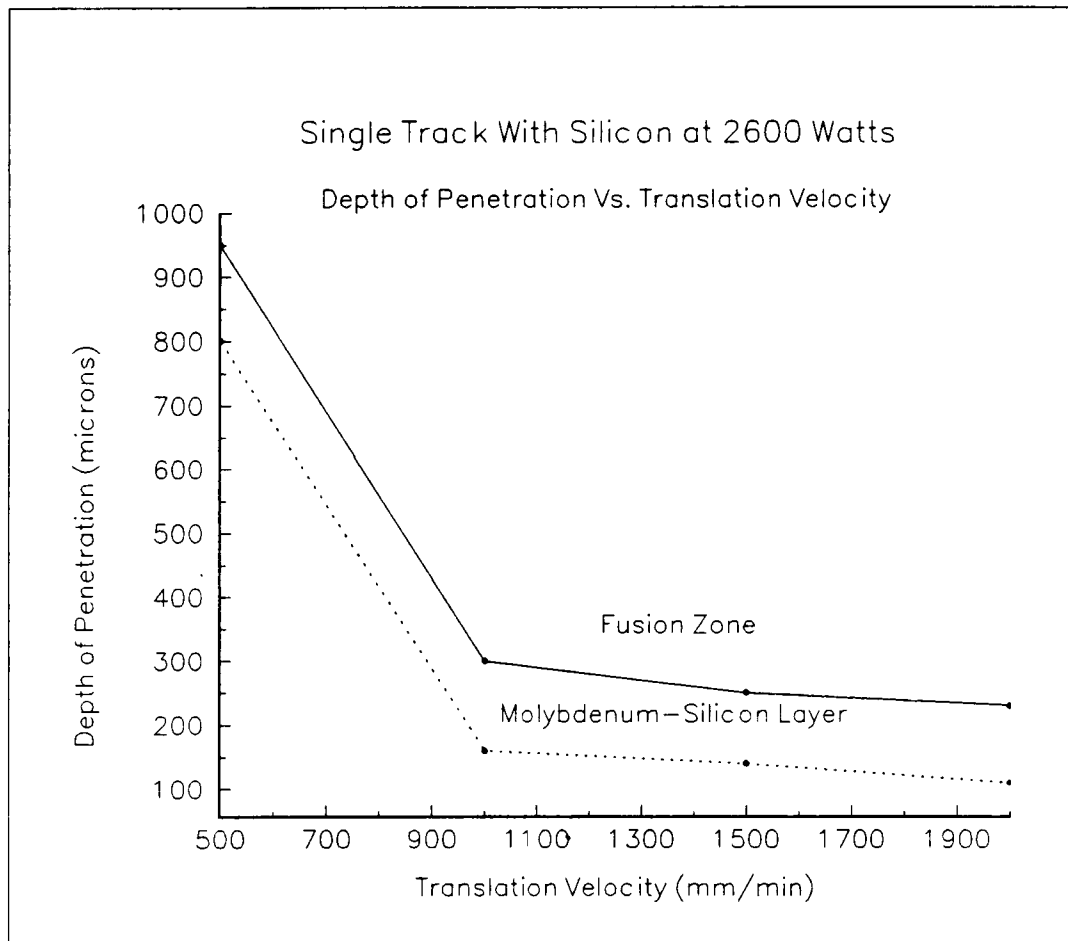


Figure 4.8 Graph of Depth of Penetration vs. Translation Velocity of the Fusion Zone and the Molybdenum-Silicon layer, for the single track with silicon at 2600 watts and a powder feeder stand off distance of 2.2 cm.

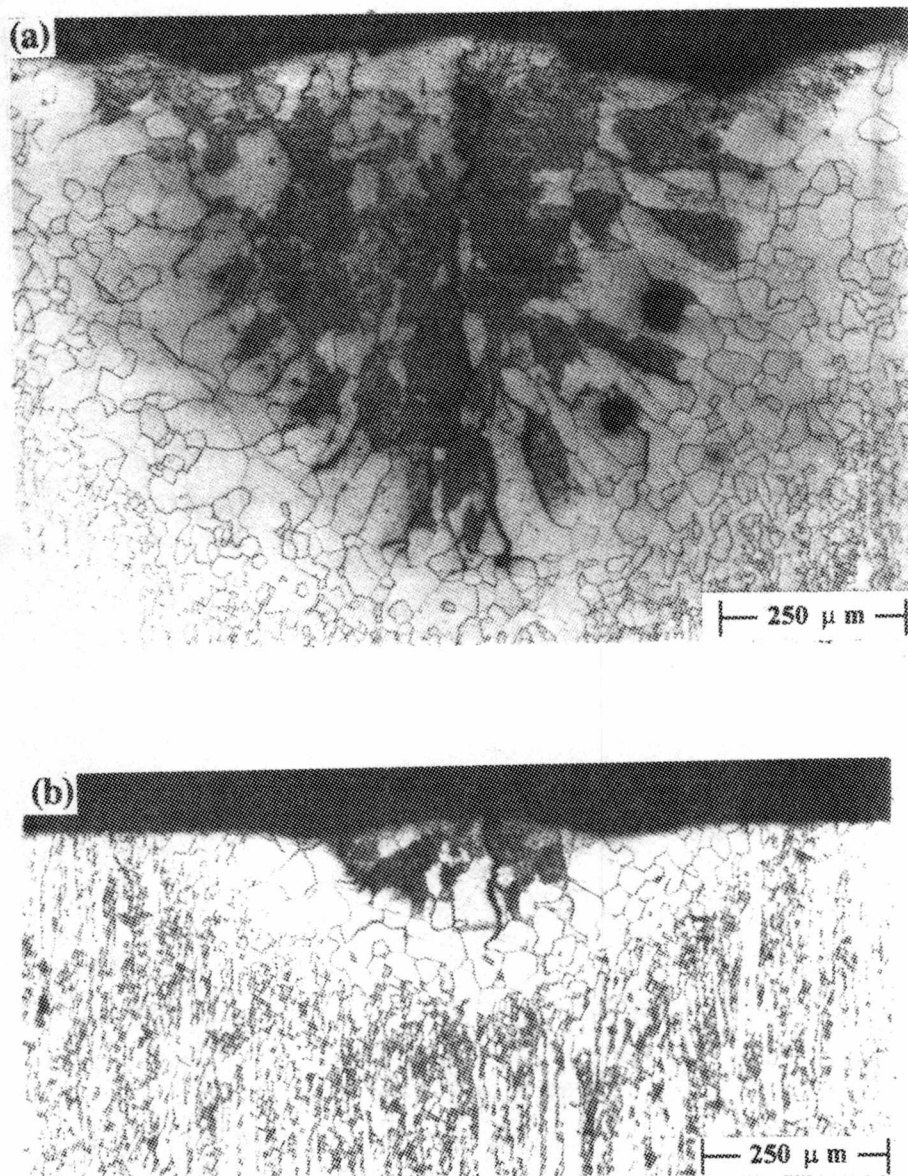


Figure 4.9 Optical photograph of a single track with silicon at a stand off distance of 2.2 cm and a translation velocity of (a) 500 (mm/min) and (b) 1000 (mm/min).

translation velocity of 500 (mm/min), a delivered laser power of 2600 watts and a powder feeder nozzle stand off distance of 2.2 cm, the width of the fusion zone was approximately 1900 μm , a region much too large for these experiments. With translation velocities of 1000 and 1500 (mm/min), a delivered laser power of 2600 watts and a stand off distance of 2.2 cm, the widths of the fusion zones were approximately 990 and 800 μm , respectively. For the translation velocity of 2000 (mm/min), a laser delivered power of 2600 watts and a stand off distance of 2.2 cm, the fusion zone was small, however, it contained a virtually unnoticeable amount of silicon. The smallest fusion zone with a small molybdenum-silicon concentration, occurred with the translation velocities of 1000 and 1500 (mm/min), these were used in the remainder of the experiments.

The idea of the multiple track with 50 % overlap with silicon is to develop a crack-free, homogenous layer of the molybdenum-silicon mixture over the molybdenum surface. In these multiple track with silicon experiments, an overlap of 210 μm , (determined from previous experiments), for the 50 % overlap, with translation velocities of 1000 and 1500 (mm/min) and delivered laser power of 2600 watts was used. These experiments formed an even molybdenum-silicon layer on the molybdenum sample with depths of approximately 100 μm , for both velocities. Photographs of these regions are shown in Figure 4.10. Microprobe analysis determined that the atomic weight percentage of silicon was less than 0.19 % in these regions. These regions also contained transverse cracks throughout the fusion zones.

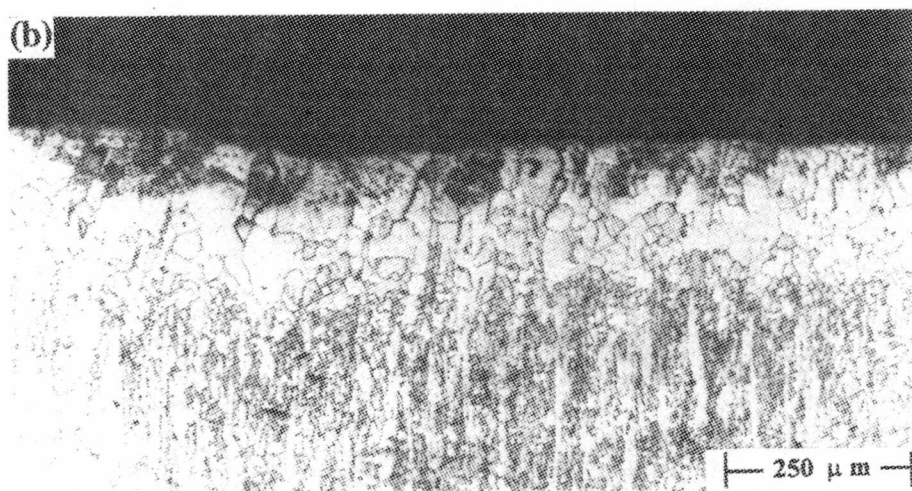
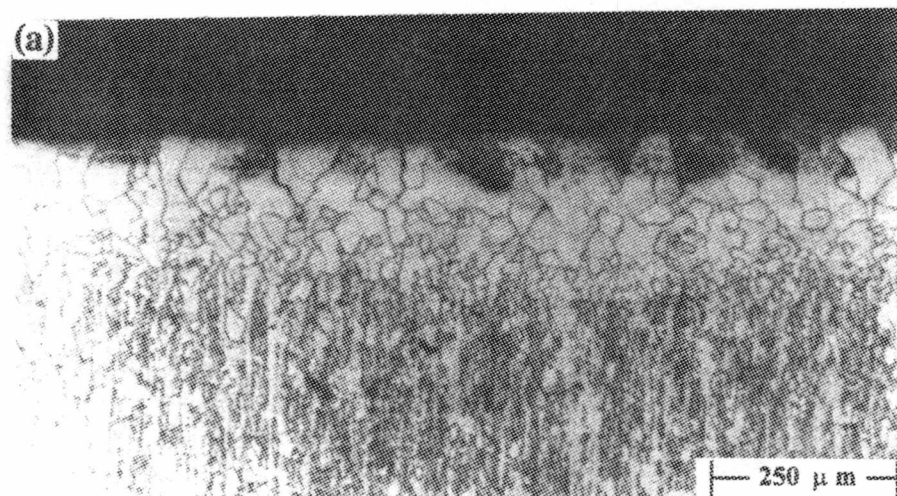


Figure 4.10 Optical photographs of multiple tracks with silicon at a stand off distance of 2.2 cm and a translation velocity of (a) 1000 (mm/min) and (b) 1500 (mm/min).

4.3) Results from the single track silicon cladding with remelting track experiments

This series of experiments was to form a silicon cladding layer on the molybdenum surface, and then reprocess this region with a wider, lower power laser beam that covers more area. The wider laser beam can remelt not just the silicon layer but also the surrounding molybdenum material and induce mixing of the two components. This process was successful in the way of producing more of a mixed molybdenum-silicon region compared to the majority of the previous experiments, however, there was no trace of the desired MoSi_2 found. Results from these experiments are give in Table 4.3. The inconsistency in the depths of the molybdenum-silicon regions for some of these results are believed to be due to the irregularity of the output of the silicon powder feeder.

Analysis of the molybdenum-silicon regions in these experiments shows that for the case of initial track translation velocities of 1000, 1500 and 2000 (mm/min), remelting track translation velocity of 1000 (mm/min) and a remelting track laser power of 1040 watts, large dendritic regions formed. Scanning electron analysis with a microprobe of these regions shows that they contain approximately 7 wt. % silicon. A photograph of one of these regions is given in Figure 4.11.

Microprobe analysis of the molybdenum-silicon regions with depths under $330\text{ }\mu\text{m}$ showed that they contain concentrations of approximately 10 % silicon, which also contain dendrites. In particular, for the case of initial track translation velocity of 2000 (mm/min), remelting track translation velocity of 1000 (mm/min) and with a

Table 4.3 Results from the single track silicon cladding and remelting track, with an initial track delivered laser power of 2600 watts, initial track beam focus 2 mm above the surface and remelting track beam focus 4 mm above the surface.

Translation Velocity _{IT} (mm/min)	Translation Velocity _{RT} (mm/min)	Laser Power _{RT} (watts)	Mo-Si depths (μ m)
1000	500	1040	1110
1500			190
2000			200
1000	500	1560	140
1500			1150
2000			330
1000	1000	1040	1090
1500			850
2000			950
1000	1000	1560	160
1500			770
2000			300

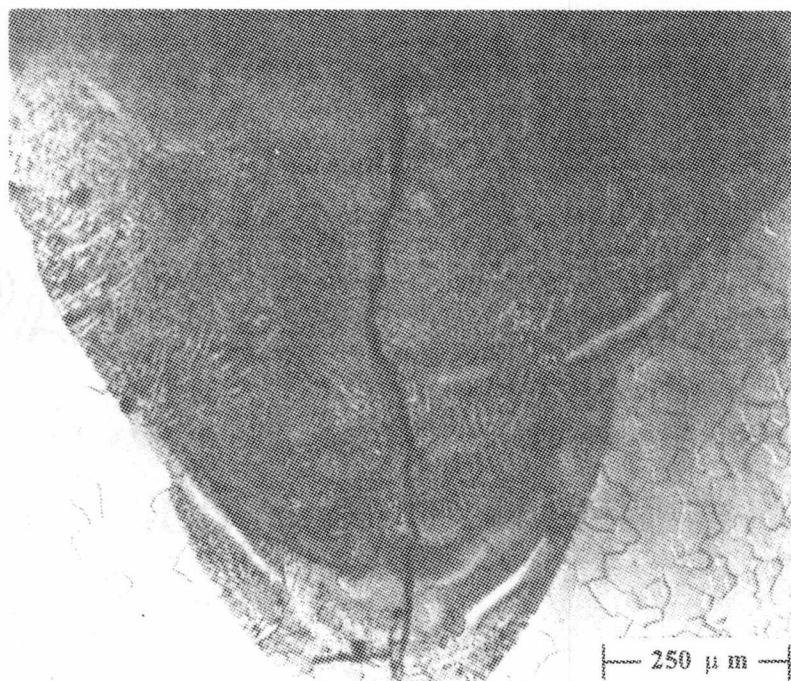


Figure 4.11 Optical photograph of a 7 % silicon region formed by initial track: 1) delivered laser power of 2600 watts, 2) translation velocity of 2000 (mm/min), 3) 2 mm beam focus and with remelting track: 1) translation velocity of 1000 (mm/min), 2) delivered laser power of 1040 watts and 3) 4 mm beam focus.

remelting track delivered laser power of 1040 watts, the molybdenum-silicon region contained three distinct layers containing different silicon concentrations. Figure 4.12 is a secondary scanning electron image, taken using the microprobe that shows these layers.

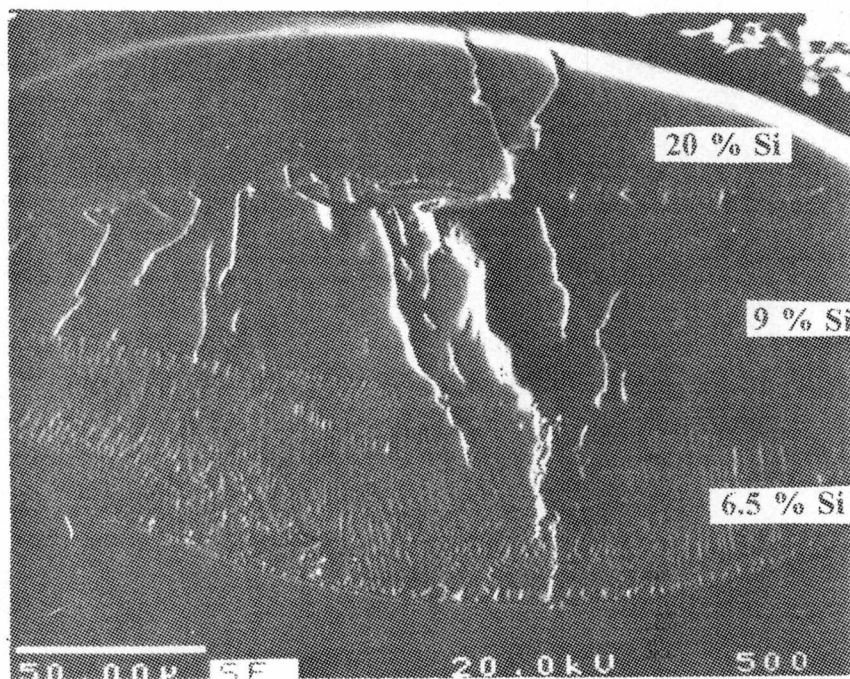


Figure 4.12 Secondary scanning electron image showing three layers of a molybdenum-silicon region formed by initial track: 1) delivered laser power of 2600 watts, 2) translation velocity of 2000 (mm/min), 3) 2 mm beam focus and with remelting track: 1) translation velocity of 500 (mm/min), 2) delivered laser power of 1560 watts and 3) 4 mm beam focus.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

It has been shown in this thesis that a molybdenum disilicide can be formed on the surface of molybdenum. This layer can be formed by adding silicon into the molybdenum melt pool, created by irradiating the surface of molybdenum with a CO₂ laser. It also has been determined that the composition of the molybdenum-silicon layer is dependent on the amount of silicon being applied to the melt pool. If too much silicon is added, cladding of the silicon on top of the surface of the molybdenum occurs. The translation velocity of the CO₂ laser beam also determines the concentration and size of the molybdenum-silicon region. At low translation velocities of 500 (mm/min), the effected regions of the sample can be as much as three times as large compared to the translation velocities of above 1000 (mm/min). However, at translation velocities of 2000 (mm/min) the concentration of the silicon in the molybdenum-silicon layer can be too low to form the molybdenum disilicide layer.

In forming these molybdenum-silicon layers, cracks appeared throughout the region. These cracks only appeared when the silicon was introduced into the melt pool of the molybdenum and mainly occurred in the molybdenum-silicon region. Throughout the experiments performed for this thesis, the occurrence of cracks in the molybdenum-silicon layer remained a challenging problem and was never resolved.

These cracks have been a common occurrence and troublesome problem for many researchers in this particular matter.

The cracks may be due to the difference in the coefficient of thermal expansion of the molybdenum disilicide and the molybdenum, $8.5 \times 10^{-6} \text{ K}^{-1}$ and $5.8 \times 10^{-6} \text{ K}^{-1}$, respectively⁹. One recommendation to alleviate the occurrence of cracks, is to add a "buffer" material to the silicon so that these two compound's coefficient of thermal expansion would be closer together. However, this buffer material should not diminish the advantages of the MoSi_2 region. One such possibility is the addition of carbon in the MoSi_2 layer. In previous work in Los Alamos National Lab, Los Alamos N.M.¹⁴, the addition of 2 wt. % carbon in MoSi_2 improved the high temperature hardness and toughness of the compound. This carbon addition can also improve the low ductility characteristic of the MoSi_2 compound¹⁴.

Another recommendation is, instead of creating molybdenum disilicide by adding silicon into the molybdenum's melt pool, but to create a small melt pool and add MoSi_2 to it, instead of silicon. The melt pool could create a bonding between the MoSi_2 and the base material, forming the MoSi_2 layer desired. Since the melt pool would be small, yet large enough to begin the mixing of the MoSi_2 and the base material, the heat affected zone should be small.

There have been inconsistencies in the depths of the molybdenum-silicon layers. This could be the result of the irregular output of the silicon powder feeder. The output of the silicon powder was sometimes in a sporadic and random amounts. Since the depths of the affected zones have been shown to be dependent on the amount of

silicon present in the melt pool, the irregularity of the silicon output appears to be a questionable parameter. Therefore, it is recommended to develop a technique to deliver the silicon powder into the melt pool in a more consistent and regulated manner.

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APPENDICES

APPENDIX A

Physical Properties for Molybdenum (Mo)^{1,15,16}

PROPERTY	VALUE
Atomic Number	42
Atomic Weight	95.94
Boiling Point (Vaporization Temp.)	4639 °C at 1 ATM
Coefficient of Thermal Expansion	4.9 $\mu\text{m}/\text{m}^\circ\text{C}$ from 25 °C to 105 °C
Crystal Structure	Body Centered Cubic $a_0 = 3.1468 \text{ nm}$ at 25 °C
Density	10.22 Mg/m^3 at 20 °C
Electrical Conductivity	34 % IACS at 0 °C
Melting Point	2623 °C at 1 ATM
Specific Heat Capacity	0.251 (J/g-K) at 25 °C, P = 100 kPa
Thermal Conductivity	0.54 (W/cm-°C) at 0 °C
Thermal Diffusivity	1.39 (cm^2/s) at 0 °C
Vapor Pressure	1.0 ATM at 4804 °C

APPENDIX B

Physical Properties for Silicon (Si)^{1,15}

PROPERTY	VALUE
Atomic Number	14
Atomic Weight	28.085
Boiling Point (Vaporization Temp.)	3280 °C at 1 ATM
Coefficient of Thermal Expansion	Linear: at 20 °C, 2.8 to 7.3 $\mu\text{m}/\text{m}\cdot\text{K}$ 2.9 $\mu\text{m}/\text{m}\cdot\text{K}$ from 0 °C to 1400 °C
Crystal Structure	Face Centered Cubic, A4; $a_0 = 0.357 \text{ nm}$ at 25 °C
Density	2.330 Mg/m^3 at 20 °C
Electrical Resistivity	1 $\text{n}\Omega\cdot\text{m}$ at 0 °C
Melting Point	1410 °C at 1 ATM
Specific Heat Capacity	677.9 (J/kg-K) at 20 °C, P=100 kPa
Thermal Conductivity	83.68 (W/m-°K) at 20 °C

APPENDIX C

Physical Properties for Molybdenum Disilicide (MoSi_2)^{1,12,17}

PROPERTY	VALUE
Atomic Weight	152.11
Coefficient of Thermal Expansion	Linear, 7.1 - 108.8 $\mu\text{m/m-K}$ at 20 °C to 1500 °C
Crystal Structure	Body-Centered Tetragonal
Density	6.2 Mg/m^3 at 20 °C
Electrical Resistivity	370 $\text{n}\Omega\text{-m}$ at 20 °C
Melting Point	2080 °C at 1 ATM
Specific Heat Capacity	420 J/kg-K at 20 °C
Thermal Conductivity	0.07 - 0.075 $\text{cal}/(\text{sec-cm}^2)(^\circ\text{C/cm})$ at 68 °F

VITA

Robert Amos Waldrop was born on November 21, 1963 in Huntsville, AL. He was the sixth child from a family of six children. He attended Davis Hills Elementary and Middle Schools and graduated from Johnson High School in June 1982. After years of fun and "hangin out", in February 1991 he received a Bachelor of Science degree in Mathematics with a minor in Physics, from Athens State College, Athens, AL. Continuing his education, he received a Bachelor of Science degree in Physics from Athens State College, in December 1992. He spent two memorable years at The University of Tennessee Space Institute, Tullahoma, TN and received an Master of Science degree with a major in Engineering Science in May 1995. He is anticipating an exciting career in engineering and research.