FABRICATION OF TARGETS TO SUPPORT
LASER-DRIVEN SHOCKWAVE EXPERIMENTS

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FABRICATION OF TARGETS TO SUPPORT LASER-DRIVEN SHOCKWAVE
EXPERIMENTS

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Prepared by J. D. Stein

Methods are being examined to fabricate and characterize precise multiple-stepped foils. Physical vapor deposition of metals onto substrates using precise masking to define each step was evaluated. A process for depositing metal onto preetched substrates to replicate precise steps is being developed.

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SUMMARY

Laser-driven shockwave experiments at Lawrence Livermore National Laboratory (LLNL) require fabrication of very small targets that may be 100 or more times smaller than those used in conventional shockwave experiments. Techniques available at Bendix Kansas City were examined to determine if satisfactory targets could be fabricated.

Prototype targets were fabricated using metal deposition and mechanical masking to build the required steps. Copper and Kapton substrates were used as supports for the deposition. A chemical etch process was used for copper substrate removal and an oxygen plasma method was examined for Kapton substrate removal. The oxygen plasma method caused excessive distortion and incomplete Kapton removal. Steps were fabricated on freestanding foil followed by bonding to copper washers. Scanning electron microscope (SEM) evaluation indicates that edge definition and spacing of steps can be achieved by metal masking; however, mask precision, mechanical damage, and distortion may be difficult to control. Replication techniques using etched silicon and glass were examined as a way to accurately control the stepped target dimensions. Test results indicate that step precision can be achieved using replication from etched glass.

Tests were completed to fabricate functional targets using copper, Kapton, and glass as substrate materials to build the detail steps. Mechanical masking provided precise steps using spectrographic slits, and machined shim stock provided some satisfactory steps. Currently, surface quality is best when Kapton is used as the substrate; however, etching development is not complete for the replication technique. Copper appears to produce a rough surface. Further investigation is required to determine the cause of this problem.

Target characterization methods are being examined using non-functional targets. Because density measurements are expected to be difficult, foil standards are being constructed to support a beta-backscatter measurement method for aluminum and an SEM measurement method for gold.

The replication process has been selected as the prime fabrication method for continued development. Inherent advantages of the replication process include the basic photolithographic techniques for precision, the adherence of aluminum to gold (eliminating the need for a chrome flash), and a single aluminum deposition run (eliminating oxide interface at the step).

DISCUSSION

SCOPE AND PURPOSE

High-power laser-generated shockwaves offer the potential of achieving unprecedented high pressures at Lawrence Livermore National Laboratory (LLNL). However, a number of technical problems must be solved before this technique can be exploited fully in accurate and useful experiments. One of the foremost problems is developing the capabilities to fabricate and accurately characterize laser-shockwave targets. The parameters of present high-power lasers demand the use of targets which may be 100 times or more smaller than those used in conventional shockwave experiments. Thus, use of existing technology for fabrication and characterization of conventional shockwave targets for laser experiments appears unlikely. Instead, techniques most likely will have to be developed or borrowed from other areas of technology.

The current target needs are for Janus experiments only. As higher energy laser systems are used, target requirements will change. However, the techniques required for Janus targets also will be needed for targets presently envisioned for long-term future programs. Therefore, the present target effort is an early step in a long range program. The reported activities were completed between January and April 1980, and work continues.

PRIOR WORK

Although no prior work specifically on fabrication of flat laser targets had been done at Bendix Kansas City, metal deposition on polyimide¹⁻³ and microelectronic technology served as a basis for initial fabrication approaches. Density characterization by beta transmission⁴ uses a previously-developed experimental apparatus and procedure.^{5,6}

ACTIVITY

Target Description

Several different types of shockwave experiments are planned. For a typical target (Figure 1), surface finish, thickness, and density are critical to successful manufacture. In experiment application, an intense laser-generated shockwave is passed through a foil of one material. The emergence of the shock from the back surface of the foil is measured by using a fast-streaking camera to record the light emitted from that surface. Because the shock heats material to extremely high temperatures

Figure 1. Janus Laser Target

(10^4 to 10^6 °C), the back surface lights up brilliantly as soon as the shock arrives. By recording the time of the onset of this bright emission, the arrival time of the shock at that surface is measured. The shock velocity is obtained by building a step of material of known height on the rear surface and measuring the arrival time of the shock at both the bottom and the top of the step. Shock velocity equals the difference in arrival times divided by step height. Because shock velocity must be measured to better than 10 percent accuracy, step heights and transmit times must be measured with even greater accuracies. Often knowing the shock velocity at several depths in the target is important. In this case, multiple-step targets will be required.

Materials used for target fabrication will include gold, copper, tantalum, aluminum, tungsten, and silver. In all cases, the base foil thickness is expected to be from 15 to 20 μm thick and must be measured to better than 10 percent accuracy. The step heights are expected to be 4 to 6 μm and must be measured to better than 1 percent accuracy. Step sharpness is critical; all surfaces should be parallel to within 1° and flat to better than 0.5 μm over a 50- μm surface. Density is especially important and should be accurately characterized for each target.

Mechanical Masking Using a Polyimide Substrate

Different techniques using a polyimide (Kapton) to provide the initial substrate for deposition of the 20- to 25- μm -thick aluminum layer and then various methods of fabrication from that point on were investigated.

The first approach involved deposition of 20 to 25 μm of pure aluminum by high vacuum evaporation from an electron beam (EB) gun source onto the shiny side of the polyimide sheet material. Small (50.8 by 50.8 mm) pieces of this polyimide/aluminum combination were then mounted in a fixture (Figure 2) that provided a 50.8- μm -thick mechanical mask for masking the next layer of aluminum. This fixture was mounted in the EB gun evaporation system, the 5 μm of aluminum was deposited, and the fixture was removed. A new mask was inserted to provide masking for the next layer. That layer is 5- μm -thick gold; however, to establish processing techniques, copper was used in place of the gold. Next, the fixture was mounted in a dc sputtering system and, finally, the 5 μm of copper was deposited. This approach provides a sheet of polyimide with a 20- μm layer of aluminum with a 5- μm aluminum step and a 5- μm layer of copper. Using a 12.7-mm-diameter punching tool, specimens with the metal steps centered are punched from this sheet. Using cyanoacrylic acid adhesive, these specimens are bonded to 10-mm-outside diameter by 6-mm-inside diameter copper rings. The next step involves using a laser to strip a 1397- μm -diameter hole in the polyimide surface to provide a pure aluminum surface (Figure 3).

No complete targets were fabricated by this technique, but targets were fabricated up to the laser stripping operation. A laser was not available for stripping; however, preliminary evaluation using a CO_2 laser indicated a selected area of polyimide could be stripped.

A second technique to fabricate the laser targets is basically the same as the first, except that the polyimide is completely removed from the 20- to 25- μm -thick vapor-deposited aluminum by a glow discharge oxidation process. This process leaves a free-standing, 20- to 25- μm -thick aluminum foil on which the various layers are deposited and the copper ring is bonded. Several complete targets were fabricated with some success.

Development effort to fabricate functional laser targets using Kapton as the original substrate material yielded six target strips, each 12.7 by 50.8 μm long. Two of these six strips were fabricated without removing the Kapton to evaluate edge definition. The other four strips were fabricated on freestanding aluminum foil obtained by removing the Kapton with a plasma oxidation process.

Figure 2. Masking Fixture Used With Kapton Substrate

Figure 3. Target Fabricated Using Kapton Substrate

Various techniques were used on these six strips to evaluate edge acuity. Two strips of pure foil used a beryllium-copper (Be-Cu) spring to hold the 38.1- μm -thick mask in place. Conventional mask fixturing was used on the remaining freestanding foil and on the Kapton-backed foil. The best deposited film edge resolution (Figure 4) was achieved using the Kapton-backed foil. This edge represented the best results to date. The other edges associated with this activity were less than satisfactory.

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Figure 4. SEM Photographs of Step Definition, S2-2

The metal masks used were machined 50.8- μ m steel foil (shim stock) mounted on top of the 20- μ m aluminum foil. A flat plate with alignment pins was used to position the masks. A separate mask approximately 100 μ m wider is used to mask the gold layer (Figure 5). Alignment and positioning have not been adequate to achieve the repeatability required for the targets. Development effort has resulted in an investigation of mechanical masks and spectrographic slit jaws to obtain good edge definition (Figure 6). Buckbee-Mears, St. Paul, MN, is constructing 25 density standard masks. These bimetallic masks are composed of 127.0- μ m Be-Cu with a 12.7- μ m nickel overplate. These masks are expected to give the edge resolution required for the density standards associated with laser target fabrication.

This investigation has exposed many problems, such as mask alignment, handling, and deposit edge definition. The targets fabricated to this point all have one or more of these problems and basic techniques are needed to overcome them. However, fabricating laser targets by one of the techniques or a modification of these techniques appears feasible.

Mechanical Masking Using Copper Substrates

Predrilled copper discs (1.25-cm in diameter) were investigated as substrates. One approach involved bonding commercially available 20- μ m-thick aluminum foil to the predrilled copper substrate, polishing the foil to an acceptable surface finish (~ 0.02 μ m), and then vapor depositing the aluminum and gold steps, respectively. The second approach involved preparing a highly polished copper surface, vapor depositing the aluminum background (~ 20 - μ m thick), and then vacuum depositing the required aluminum and gold steps. The above procedures were used according to the sequence listed in Table 1.

Mechanical masks were used for fabricating thin metal steps on the polished copper discs (Figure 7). Polishing procedures were developed which yield a flat, highly polished copper surface onto which aluminum and gold were deposited.

Ten samples were processed through gold deposition. Four samples were rejected because of step position. Six samples were selected for copper removal; however, gold delamination was evident on two samples. Characterization was completed for four samples, and a poor surface finish (Figure 8) was observed on all surfaces. This condition is possibly related to the copper preparation, and suitable lapped surfaces will require electropolishing.

TOOLING PIN REGISTRATION

MASK LIFTING OFF ALUMINUM FILM

Figure 5. Alignment of Mechanical Masks

Replication Method

Concepts to fabricate targets included a method of replicating steps etched into a suitable substrate (Figure 9). Photolithographic methods common to the microelectronics industry were believed to be compatible if the substrate could be etched away to leave access to the target area.

Techniques for the back side hole etching progressed well. Both hard glass and silicon samples were etched, with the silicon

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Al

Figure 6. SEM Photographs of Step Definition,
Spectrographic Slit Jaws

Table 1. Mechanical Masking Fabrication Techniques

Method 1	Method 2
Predrill copper discs leaving 254- μ m wall thickness	Predrill copper discs leaving 254- μ m wall thickness
Bond aluminum foil to undrilled surface	Polish the undrilled surface (0.01- μ m)
Polish aluminum surface ($\sim$ 0.02 μ m)	Vapor deposition of aluminum (20- μ m-thick)
Vapor deposit aluminum step (5- μ m-thick) with mechanical masking	Vapor deposition of aluminum step (5- μ m-thick) with mechanical masking
DC sputter gold step (5- μ m-thick) with mechanical masking	DC sputter gold step (5- μ m-thick) with mechanical masking
Etch hole in copper disc (from back side)	Etch hole in copper disc (from back side)

preferred. Standard photoresist masks did not maintain their integrity for the required long etch periods. Therefore, a combination of gold (photo defined) and wax masking was used. Other materials will be investigated. The near vertical sides in the etched silicon suggests that well defined edges (under 40°) are possible in the replication approach (Figure 10).

Silicon was intended to be the primary substrate material in the replication process. However, problems occurred with the formation of a gold-silicon eutectic and in the wet process etching of the steps. The first problem was solved by holding down the evaporation temperature, but irregular etching has no easy solution, according to personnel at Sandia National Laboratory Albuquerque (SNLA). They advised that the solid-state industry has spent a great deal of effort on etching silicon and that with elaborate equipment and tight controls, smooth controlled surfaces can be obtained. They suggested reactive ion etching, ion milling, or a change in substrate material from silicon to glass.

Two glass samples were etched in a 1:1:1 solution of hydrofluoric, nitric, and acetic acids. Surface profilometer measurements of

Figure 7. Mechanical Masks Used With Copper Substrate

the etched 7059 glass revealed relatively rough surfaces. However, SEM photomicrographs on etched microscope cover glass showed extremely smooth surfaces. Etching of this softer glass is relatively fast, about 250 nm/s, and further development may be necessary on the etchant. However, steps generated in the microscope glass material look good (Figure 11).

The appearance of a step in the back side of the target (laser side) was thought to be caused by tension in the films at right angles to the step. This tension may be caused by the difference in coefficient of contraction between the substrate materials and the metals deposited. Targets were constructed in strip form parallel to the step so that these deforming forces would be at a minimum.

The first series of replicated targets was completed and analyzed. The replication approach shows considerable promise, especially in step definition and precision. Some problems, however, must be overcome before good targets can be fabricated on a routine basis. Two problems were especially evident in the first series generation.

Figure 8. SEM Photographs of Step Definition, Sample Number 5

Figure 9. Fabrication of Janus EOS Target, Replication Method

- The glass substrates were too small and too fragile to survive handling and lapping. New glass which is both larger in size and thicker has been obtained.
- In back side etching the hole through the substrate, the etchants attack the aluminum layer. A stop layer of chrome-gold in the next run should improve this situation. Other stop layers also will be investigated.

The stop layer material evaluation includes the use of chrome-gold (Series II) and chrome-copper (Series III). The first two

1500X

Figure 10. SEM Photograph of Step Definition, Silicon Substrate

substrates (each containing 12 possible targets) in the Series II fabrication run are in process using the chrome-gold, glass-etch mask material. In this configuration, chrome-gold is used not only as mask material, but a stabilized protective layer also is deposited in the etched two-step channel beneath the 5- μ m-gold step and the 20- μ m-aluminum deposition. Preliminary tests indicate that successive cerium-ammonium-nitrate and potassium-iodide/iodine etch steps are necessary to remove the layer. Neither etchant readily attacks aluminum. Series III target fabrication was initiated also. This set of targets was to be fabricated with the chrome-copper, glass-etch masking, but the chrome underlayer did not etch after the copper was removed. Investigation is continuing to select compatible stop layer materials and substrate etchants.

Characterization

Targets fabricated using mechanical masking have been used for initial characterization. Scanning electron microscope evaluation was used extensively to select suitable nonfunctional targets for specific measurements. Thickness measurements were made by surface profilometer (Clevite), Zeiss interferometer, and by contact interferometer. Two samples deposited on copper (0 and 5) and three 50.8-mm-long strips (S2-2, S1-2, and S1-1) deposited on Kapton were measured, and the data are summarized in Table 2. Calibration of equipment and improved target fabrication are expected to provide satisfactory dimensional characterization.

Table 2. Dimensional Data Obtained From Nonfunctional Targets

	Sample				
	0	5	S2-2	S1-2	S1-1
Aluminum Foil Thickness (μm)	17.8	19.1	22.6	25.9 to 28.4*	26.1 to 29.3*
Aluminum Step Thickness (μm)					
Clevite	4.06	**	5.72	5.72	6.35
Zeiss	3.81	**	5.54	4.83	***
Gold Step Thickness (μm)					
Clevite	4.57	**	6.35	8.89	8.26
Zeiss	4.98	**	5.69	***	***
Aluminum Step Width (μm)	47	-	75	75 to 100	75 to 100

*Thickness range of strip sample.

**Total by Clevite 10.2 μm ; total by Zeiss 10 μm .
Witness sample 4.3 μm aluminum; 6.0 μm gold by Clevite.

***Sample not sufficiently flat for precise measurements.

Attempts to determine density of deposited aluminum and gold by weight and volume have been difficult because of the uncertainty of volume measurements. A sample of aluminum foil provided by LLNL and supplied by Reactor Experiments Inc., San Carlos, CA, was evaluated. The weight of 0.710646 and volume of 0.25836 cm^3 corresponds to a density of 2.75 g/cm^3 . Reactor Experiments Inc. was contacted and could not offer an immediate explanation for the high density value; however, a water displacement technique is used at their plant and they agreed to investigate the possibility of providing samples with density values to Bendix.

Nondestructive method development, such as beta transmission or backscatter for aluminum and SEM for gold, have been delayed because of standards fabrication. A review has been initiated to determine if density measurements can be made that relate to fundamentals such as absorption coefficient and that allow the application of nondestructive methods prior to rigorous standard

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1000X

GOLD FROM
INCOMPLETE ETCH

500X

Figure 11. SEM Photographs of Step Definition, Glass Substrate

certification. A literature search has been initiated and several experimental arrangements have been discussed. The current thinking is that energy transmission can be used to assign values to materials that can then be used to support techniques such as backscatter for actual targets. Initial experiments will be conducted to select energy source, detector, and apertures.

Preliminary measurements of two foil samples indicate that the beta transmission technique has good sensitivity for density variations, on the order of 2 percent. These measurements were made using essentially an uncollimated beam. Whether this method is effective for a tightly collimated beam/detector system remains to be shown. Functional designs are being developed for a source/detector configuration that might be capable of making measurements on the actual step of interest.

ACCOMPLISHMENTS

Readily available methods to fabricate laser targets were evaluated against product requirements. Prototype targets were fabricated using metal deposition and mechanical masking to build required stepped thicknesses of aluminum and gold. A micro-electronic technique based on metallized replication of detail preetched in silicon or glass was examined and selected for continued development. Product requirements have not yet been met; however, the prototype parts indicate feasibility and provide a basis for specific development.

FUTURE WORK

Development associated with the laser target activity includes improved mechanical masking. Special tooling, to be designed, is anticipated for mounting on a continuous cable processor. This tooling would provide the capability to deposit the aluminum and gold steps within the high vacuum deposition chamber sequentially without opening to atmosphere. Manipulators with micrometer-type resolution would be used to position mechanical masks as required.

The major development required for the replication method involves the selective etching of glass from the aluminum and gold target steps. The functionality of the target without glass removal will be evaluated by LLNL. Careful (drop depletion) techniques to remove the glass also will be evaluated at LLNL. An on-going search will be maintained to provide simple substrate removal. Additional efforts to improve the targets will include:

- Investigating different etchants, etching techniques, and substrate materials for improved master generation;
- Refixturing the vacuum chamber for improved uniformity and deposition control; and
- Making process improvements to ensure that residual gold is not left in the master at the aluminum step.

Beta transmission for aluminum density experimentation will include the use of Lamberts Law in the form of

$$\ln (I/I_0) = -\left(\frac{17}{E_m^{1.14}}\right)\rho t .$$

Source energy studies using sample foils will be tested to determine if density analysis is feasible.

Scanning electron microscope methods will be developed for gold density based on carefully constructed density standards. Dimensional characterization is expected to be satisfactory after measurement calibration and improved target fabrication.

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