Impact of Film Stress and Film Thickness Process Control on GaAs-TiAu Metal Adhesion

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The fabrication of GaAs-based optoelectronic ridge-waveguide devices requires deposition of a topside-contact metallization for proper device operation. Fabrication delays occurring during the processing of TiAu-contact pads have been linked to poor adhesion and metal blister formation, factors that negatively affect the final device yield. In this study, we examined sputter-deposited Ti and Au films to determine the impact of film-thickness process control and film stress as measured by wafer bow. We theorized that competing stress relaxation forces between the Ti and Au films would produce a post-deposition change in wafer bow, which affects the Au film, setting the stage for blister creation. We now report the development of a reduced-stress sputter-deposited TiAu-contact metallization and demonstrate the utility of the modified process with fabrication of blister-free ridge-waveguide devices with high device yield.

Key words: GaAs, film stress, metal blister, blister, adhesion, device yield

INTRODUCTION

The fabrication of GaAs-based ridge-waveguide optoelectronic devices requires the deposition of a topside-contact metal. Optimized metal film composition is a function of semiconductor contact layer conductivity, as well as device design.1 Two suitable non-alloyed-contact metals for highly doped p-type GaAs are TiAu and TiPtAu. In this study, a sputter-deposited TiAu (50-nm Ti/200-nm Au) metallization was used as the contact metal for GaAs-based slab-coupled optical waveguide (SCOW) laser devices which have a (> 5 × 1019) p-type-contact layer. The metal deposition process included an in situ sputter etch to promote a clean semiconductor–metal interface with enhanced adhesion.2,3 The initial Ti layer provided adhesion enhancement while also functioning as a barrier between the Au layer and GaAs surface, eliminating undesirable Ga-Au interactions. The top Au layer provided an oxide resistant electrical contact suitable for probe testing, wire bonding, and solder-based packaging. The contact-metal film was sputter-deposited on a pre-cleaned photoresist-free sample surface, creating a continuous metal sheet, to ensure optimal adhesion. A photolithographic process was subsequently used to delineate contact-metal pads, and the removal of excess deposited Au and Ti was achieved with wet chemical etching, thus isolating the devices as well as successfully completing the fabrication process. Fabricated devices must have low metal–semiconductor contact resistance (< 1 × 10⁻³ Ω cm²) and a defect-free appearance with good adhesion to ensure proper device operation and to meet packaging requirements.4,5

In our laboratory, early efforts to fabricate GaAs SCOW devices were beset by the random lot-to-lot appearance of blisters in the TiAu-contact-metal pads. Optical microscope images of a GaAs SCOW device wafer with metal contact-pad blisters is shown in Fig. 1. These adhesion failures generated concerns about the quality of the metal—
semiconductor electrical contact resistance, and ultimately, device performance and reliability. Previous published studies had linked blister formation to inadequate surface cleanliness and SiO₂ dielectric thermal instability, factors that must also be managed to optimize device yield. Moreover, a direct correlation between the appearance of blisters and contact pad processing time was also previously observed. Contact pad processing delays of less than ~24 h produced blister-free contact pads and very high device yields; conversely, the incidence of blister formation increased as a function of time when delays were greater than ~24 h. Together, these observations indicated the importance of fabrication scheduling to minimize time delays between metal deposition and contact pad processing.

A survey of thin film stress measurements was performed to evaluate various contact metal combinations used in our laboratory. This data set revealed a change in the film stress of a sputter-deposited TiAu-contact metallization over time. The measured change in film stress represented a physical change in the bow of the TiAu coated wafer, rather than a change in the stress of an individual film. This wafer bow change, in the direction of a more compressive profile, might physically compromise the top Au film which, when exposed to additional device processing and thermal cycling, could ultimately be responsible for blister formation. Our findings also indicated that the addition of Pt to the metal stack or reduction of Au thickness resulted in a bow-stable film. The determination of changing wafer bow was the basis of the current set of studies and hypothesis development.

Previous reports of thin metal film adhesion failure have examined the impact of film stress relief and surface disruptions as an adhesion-loss failure mechanism. It is also well known that the residual stress of sputter-deposited metal films can frequently be minimized by optimizing deposition parameters. Based on this information and our process observations and experience, we theorized that competing Ti and Au film stresses are the source of a post-deposition change in wafer bow which negatively impacts the deposited metal surface. Additionally, we hypothesized that modification of the TiAu sputter-deposition process to minimize wafer bow and reduce post-deposition wafer bow change should result in elimination of blister formation. To test our theory, as well as examine Ti and Au film stress as a function of deposition rate and DC sputter power, we have performed a series of test depositions using a variety of settings. The change in resultant wafer bow was minimized by selecting low-stress sputter-deposition conditions and optimizing film thicknesses. Based on these findings, we can now report the development of a reduced-stress, wafer-bow-stabilized sputter-deposited TiAu-contact metallization. GaAs SCOW devices, fabricated by utilizing this modified deposition process for topside-contact metal, were blister free and fabricated with high device yield, regardless of contact pad processing delay times.

**EXPERIMENTAL PROCEDURES**

The full GaAs SCOW fabrication sequence has been described in detail previously. A ridge waveguide was dry etched into the semiconductor utilizing an ICP-RIE process. Once the ridge was defined, the surface was coated with a 200°C low-stress Samco PECVD SiO₂ dielectric layer and annealed at 450°C for 45 s for stress reduction and improved film stability. Photolithographically patterned contact vias were etched utilizing CF₄ in a parallel plate RIE system. The sample was thoroughly cleaned and the sample surface was coated with the SCOW sputter-deposited TiAu (50-nm Ti/200-nm Au) contact metallization. The sputter-deposition system used in this study was a Denton model Discovery-18. The system was configured with three DC heads and one RF sputtering head. DC power was supplied by a pair of Advanced Energy model MDX 1.5 K power supplies. Argon was utilized as the sputtering process gas, with 25 standard cubic centimeter per minute (sccm) gas flow regulated by a mass flow controller. There was no active or passive substrate temperature regulation and no active process-chamber pressure control. Deposition parameters used to deposit 50-nm of Ti were 150 W DC and deposition time was 350 s. Parameters used to deposit 200-nm of Au were 75 W DC over a time interval of 630 s. Contact pads were defined photolithographically; excess Au and Ti were wet chemically etched utilizing metal-specific etchants (Transene Corp) to remove the unwanted metal regions. Optical microscope images of GaAs ridge-waveguide devices, captured immediately after TiAu sputter deposition (a), photolithographic processing (b), and wet chemical etching (c), are shown in Fig. 2. To complete the fabrication, the
substrate backside was thinned by mechanical lapping and chemical polishing, and a backside n-metal was deposited. The sample was dismounted and a 450°C for 45 s n-metal alloy anneal was performed. Visible contact-pad-metal blisters, if present, will appear after this anneal step.

In order to simulate the SCOW device fabrication procedure and replicate the generation of contact-pad-metal blisters on GaAs substrates, a streamlined-fabrication process was developed. The surface of two 20-mm × 30-mm GaAs wafer test samples (A, B) were coated with a 300-nm, 200°C plasma enhanced chemical vapor deposition (PECVD) SiO₂ dielectric layer, and annealed at 450°C for 45 s to reduce stress and improve film stability. Subsequently, the samples were coated with the SCOW sputter-deposited TiAu (50-nm Ti/200-nm Au) contact metallization and annealed immediately (Sample A), and 7 days post-deposition (Sample B). As demonstrated by optical microscope imaging (Fig. 3), Sample A (a) showed no sign of surface disruption or blistering, while Sample B (b) had developed numerous surface blisters. This experiment suggested that blister formation was a reproducible problem and verified past observations linking processing delays to the appearance of blisters. Surface analysis of Sample B utilizing a scanning electron microscope (SEM) is illustrated in Fig. 4. These images show the sample immediately after deposition (a), on day 7 (b), and after thermal anneal (c). There was a change in appearance at day seven as grain boundaries become more pronounced and grains appear to be grouped in clusters. The
post-anneal examination reveals the presence of cracks at the Au surface.

A series of experiments were subsequently performed to examine the SCOW device fabrication sequence and to understand the impact of Ti and Au film stress and resultant changes in wafer bow on blister creation.

**Film Stress and Wafer Bow Evaluation**

Stress and wafer bow measurements were performed utilizing a Toho model FLX-2320-S thin-film stress-measurement system. The Toho tool utilizes a laser scanning method to measure the wafer radius of curvature pre- and post-deposition. Stoney’s equation was used to calculate the film stress and wafer bow. The measured and reported film stress and wafer bow numbers are the average of two separate scans in the Toho tool: one parallel to the major wafer flat (position 0) and one perpendicular to the flat (position 90). The reproducibility and precision of stress measurement, influenced by a number of factors including sample position reproducibility, was $\pm 8\%$. The change in wafer bow is reported as $\Delta$-Bow, a comparison of wafer bow before and after a device-processing event. To determine wafer bow changes as a function of time, the data point collected immediately after deposition was used as the initial data value. Thin film stress and wafer bow were examined by depositing large-area films on $\sim 290 \, \mu m$ thick (100) silicon substrates. A survey and compilation of various sputter-deposited metal films typically used in device fabrication was conducted. Film-stress measurements, performed immediately after deposition ($t_0$) and 1-week later ($t_1$) are shown in Table I. Evaluation of the typical SCOW TiAu (50-nm Ti/200-nm Au) metallization revealed a post-deposition change in film stress and wafer bow as a function of time.

To further explore this topic we expanded our studies to include experiments, which are detailed below, aimed at characterizing the: (1) impact of contact pad processing on wafer bow, (2) assessment of Ti and Au film stress, (3) effect of film thickness on wafer bow, and (4) developing a modified deposition process.

**Contact Pad Processing**

In order to understand the benefit of timely TiAu-contact pad processing on blister formation, the impact of contact pad fabrication on wafer bow was examined. Large area SCOW TiAu-contact metal films were deposited on silicon test wafers; photolithographic patterning of the full wafer surface and wet chemical etching of Au and Ti followed. Three samples were prepared: Sample A1 was patterned with the SCOW contact-pad pattern consisting of 450-$\mu m$ wide opening stripes on 520-$\mu m$ centers, and Samples B1 and C1 were patterned utilizing 90-$\mu m$ wide opening stripes on 190-$\mu m$ spaces which were photo mask aligned parallel (B1) or perpendicular (C1) to the wafer major flat. The wafer bow was measured before and after wet chemical etching.

**Ti and Au Layer Stress Contribution to Wafer Bow**

The influence of individual Ti and Au layer stresses on the TiAu-contact metal coated wafer bow was examined by wet chemically removing the Au layer from TiAu-coated test samples. A second set of three silicon test wafers were prepared: Sample A2 was coated with a single Ti layer and...
served as a control. Samples B2 and C2 were coated with SCOW TiAu-contact metal film. Wafer bow measurements were performed on all samples. The Au layer was immediately wet chemically removed from Sample B2 using Transene TFA gold etchant, followed by wafer bow measurement. Sample C2 was monitored for 7 days, after which the Au layer was wet chemically removed and the wafer bow measured.

**Sputter-Deposition Power and Impact on Film Stress**

The effect of sputtering system deposition rate, controlled by DC sputtering power, on film stress was examined. A third set of silicon wafers were coated with single layer Ti and Au films deposited at three DC powers: 38, 75, and 150 W. Stress and wafer bow measurements of the samples were performed for 7 days.

**Ti Thickness and Wafer Bow**

A fourth set of silicon wafers were coated with various thickness Ti films to evaluate the impact of film thickness on wafer bow. Stress and wafer bow measurements were performed immediately after each deposition.

**TiAu Deposition and Thickness Impact on Wafer Bow**

The effect of individual film stress and improved thickness of Ti and Au layers on wafer bow was examined by preparing a fifth set of large area TiAu metal-coated silicon test wafers. Sputter-deposition conditions of 38, 75 and 150 W were used with a target thickness of 35-nm for Ti and 200-nm for Au. Film stress and wafer bow measurements were performed on all samples for 10 days.

**Modified TiAu-Contact Metallization Evaluation**

Based on data from the film stress and wafer bow analyses, a reduced-stress bow-stabilized TiAu-contact metal deposition process was developed. To evaluate the modified TiAu-contact metallization, two 20 × 30 mm GaAs samples (C, D) were processed utilizing the streamlined fabrication process. Sample C was immediately annealed at 450°C for 45 s and inspected. Sample D was annealed 7 days post-deposition and inspected.

As an additional evaluation of the modified TiAu-contact metallization, simple tape-pull tests were performed to assess TiAu film adhesion. Transmission Line Measurement (TLM) test structures were fabricated and measured utilizing GaAs SCOW epitaxial material to evaluate metal–semiconductor contact resistance.14,15

**RESULTS**

**Film Stress and Wafer Bow Evaluation**

**Contact Pad Processing**

Metal contact pad fabrication etch test results are shown in Table II. In each case, the patterned and etched wafers reverted to their pre-deposition bow once the TiAu-contact metal was etched, regardless of photoresist pattern orientation. This confirmed the value of timely contact pad processing as the TiAu-contact metal stress-induced wafer bow was immediately relieved by the patterning and etching processing steps.

**Ti and Au Layer Stress Contribution to Wafer Bow**

The influences of individual film stress on TiAu-contact metal coated wafer bow were examined (Table III). The data demonstrated that the initial Ti layer deforms the wafer in a compressive (+) direction, while film stress from the deposition of

### Table I. Film-stress measurement data of various sputtered-metal films typically used in device fabrication

| Metallization | Thick (nm) | Stress, $t_0$ (mPa) | Stress, $t_1$ (mPa) | %Change | Target thickness (nm) |
|---------------|------------|---------------------|---------------------|---------|-----------------------|
| Ti            | 34.8       | 2100                | 1970                | 6.2     | 50                    |
| Au            | 200.0      | 200                 | 215                 | 7.5     | 200                   |
| Ti/Au         | 87.1       | 860                 | 840                 | 2.4     | 50/200                |
| SCOW Ti/Au    | 290.0      | 90                  | 10                  | 89.0    | 50/200                |
| Ti/Pt/Au      | 170.0      | 540                 | 545                 | 0.9     | 50/100/50             |

Film-stress measurements, performed immediately after deposition ($t_0$) and 1 week later ($t_1$).

### Table II. Metal contact pad fabrication test data highlighting A-Bow before and after metal etching

| Sample                          | A-Bow (μm) |
|---------------------------------|------------|
| TiAu coated average             | 1.2        |
| Patterned and etched A1         | 0.1        |
| Patterned and etched B1         | 0.2        |
| Patterned and etched C1         | 0.2        |

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the top Au layer caused a more tensile (−) distortion. After the 7-day post-deposition period, the TiAu coated wafer bowed in a compressive (+) direction very similar to that of the Ti coated wafer. When the Au layer of Sample B2 was removed immediately after TiAu metal deposition, the Δ-Bow reverted to that of a Ti coated wafer. The Δ-Bow of Sample C2, which had experienced a bow change over time, was unaltered after Au removal and resembled the bow of a Ti coated sample. This experiment provides evidence that the titanium film dominates the TiAu coated SCOW wafer bow as a function of time.

Sputter-Deposition Power and Film Stress

Sputter-deposition conditions were analyzed to determine the impact of DC power on film stress. The film-stress and wafer bow measurements of individual films were stable as a function of time. Initial film stress was plotted as a function of DC power for Ti and Au films and results are shown in Fig. 5. A reduction in DC power resulted in a lower film deposition rate and a lower measured stress was recorded. The three Ti coated test samples were examined using SEM; a summary of grain size measurement data (Table S1) and individual images (Figs. S1–3) generated from these analyses are included in Supplementary Material. The average grain size of the 75 and 150 W depositions were similar. The average grain size of the 38 W deposition was clearly smaller.

\[
\sigma = \frac{4}{3} \times \frac{E}{(1 - \nu)} \times \frac{I^2 B}{t_f L^2}
\]

where \(\sigma\) = total film stress, \(E/(1 - \nu)\) = substrate elastic constant (\(E\) is Young’s modulus and \(\nu\) is Poisson ratio), \(t_s\) = substrate thickness, \(t_f\) = film thickness, \(B\) = wafer bow, and \(L\) = scan length. In a case where the substrate materials (substrate thickness and scan length) are constants, the equation becomes:

\[
\sigma \times t_f = C \times B
\]

where \(C\) = collected constants. The film stress-film thickness product is directly proportional to the wafer bow indicating that decreased film stress and/or thickness will result in wafer bow reduction. In addition to developing deposition conditions for reduction of individual film stress, we optimized the Ti thickness at 35-nm based on additional optimization experiments and calibrated the Au

**Table III. TiAu coated wafer test data highlighting Δ-Bow before and after Au etching**

| Sample | Metal          | Δ-Bow (µm) | Δ-Bow after Au etch (µm) |
|--------|----------------|------------|--------------------------|
| A2     | Ti             | 2.1        | 2.0                      |
| B2     | Ti\Au day 1   | 0.5        | 2.1                      |
| C2     | Ti\Au day 7   | 2.2        | 2.1                      |

Fig. 5. Deposited film stress as a function of sputtered DC power.

Fig. 6. Ti film Δ-Bow as a function of film thickness.

**Ti Thickness and Wafer Bow**

Data obtained on wafer bow for three different DC powers as a function of Ti film thickness are shown in Fig. 6; these results confirmed that film thickness directly affects wafer bow. In our applications, the ideal Ti film thickness is the thinnest possible layer that minimizes wafer bow, yet maintains functionality as an adhesion layer.

Stoney’s equation describes the total stress of a deposited film based on the wafer radius of curvature before and after film deposition. A common form of the equation relating thin film stress to wafer bow is:
film thickness to accurately deposit 200-nm to accommodate device and packaging requirements.

**TiAu Deposition, Thickness Parameters, and Wafer Bow**

The impact of lower stress deposition conditions and optimized film thickness on wafer bow were evaluated using a series of silicon test wafers. The wafers were coated with TiAu-contact metal films using a variety of deposition parameters and film thicknesses, and wafer bow was monitored for 7 days. The results of selected samples are listed in Table IV. As the individual film thickness and sputter-deposition DC power (film stress) were reduced in the TiAu metallization, the magnitude of the Δ-Bow decreased. Sample J was produced using the original SCOW TiAu-contact metallization with higher-stress deposition conditions and poor layer thickness control. Improving the individual layer thickness process control (Sample P) had a significant impact on Δ-Bow. A reduction in individual layer stress along with improved thickness process control (Sample M) resulted in further diminution of the Δ-Bow. Sample M displayed the ideal combination of reduced film stress and optimized film thickness, and was selected as the modified TiAu-contact metallization process. A comparison of the Δ-Bow as a function of time for the original SCOW TiAu-contact metallization (Sample J) and modified TiAu-contact metallization (Sample M) is displayed in Fig. 7.

The modified TiAu-contact metal process development involved determination of optimized sputter-deposition DC power parameters and target film thicknesses. All depositions were performed with an argon process gas flow of 25 sccm. There was no active or passive substrate temperature regulation and no active process-chamber pressure control. The modified Ti film deposition conditions were 75 W DC power and a target thickness of 35 nm, while the modified Au film deposition conditions were 38 W DC power and a target thickness of 200 nm.

**Modified TiAu-Contact Metallization Evaluation**

As a test of the modified TiAu-contact metallization, simulated fabrication samples were processed. The results of this analysis are shown in Fig. 8. The modified TiAu-contact metallization did not blister when processing was delayed for 7 days.

As an additional validation of the modified TiAu-contact metallization, adhesion tape-pull tests and TLM measurements were performed. All samples passed simple scotch tape adhesion tests, confirming acceptable adhesion. Contact resistance TLM test structures were fabricated utilizing SCOW epitaxial material; results showed an acceptable metal–semiconductor contact resistance in the $1 \times 10^{-6} \, \Omega \, \text{cm}^2$ range.

**Discussion**

Based on process observations and experimental results, we can propose a stress related blister formation mechanism for the original sputter-deposited TiAu-contact metallization. The Ti film, as deposited, caused the GaAs substrate to bow in a positive compressive direction. The addition of the Au film altered the GaAs substrate bow in a negative tensile direction. Processing delays that occur immediately after deposition allowed the wafer bow to change, in the direction of a more compressive profile and similar to the profile of a Ti coated wafer. This bow change over time compromised the Au surface ultimately contributing to the formation of metal blisters. Timely processing of the contact pads immediately after metal deposition relieved the stress-induced substrate bow and

| Sample | Ti DC (W) | Au DC (W) | Ti Tk (nm) | Au Tk (nm) | Δ-Bow (µm) | Summary |
|--------|-----------|-----------|------------|------------|------------|---------|
| J      | 150       | 75        | 70         | 300        | 1.48       | Original SCOW—poor thickness control |
| P      | 150       | 75        | 35         | 200        | 0.88       | Improved thickness control |
| M      | 75        | 37        | 35         | 200        | 0.65       | Reduced film stress |

Fig. 7. Temporal change in wafer bow (Δ-Bow) normalized to initial post deposition bow, for original SCOW (J) and modified (M) TiAu-contact metallization samples.
eliminated surface changes, the source of metal blisters. In the case of the original SCOW TiAu-contact metallization, the Ti film had a target thickness of 50-nm and the Au film of 200 nm. In reality, both processes yielded layers that were subsequently found to thicker by 40% due to process drift considered inconsequential at the time. This additional unintended thickness further increases the film-stress impact on wafer bow.

Sputter-deposition conditions have been developed to produce a modified TiAu-contact metallization utilizing reduced-stress optimized-thickness Ti and Au films. Device wafers coated with the modified TiAu-contact metallization experience a greatly reduced change in wafer bow when subjected to processing delays. GaAs SCOW device wafers that were fabricated with this improved process were blister free with high device yields regardless of processing delays in contact pad fabrication. The modified TiAu-contact metallization also provides process flexibility as devices can be fabricated without requiring a tight time window for contact pad fabrication steps. The elimination of contact-pad blisters during the fabrication of GaAs SCOW devices requires management of multiple factors including surface cleanliness, thermal stabilization of PECVD films and optimization of TiAu-contact pad film stress and individual layer thickness.

SUMMARY

We have developed a reduced-stress, wafer-bow-stabilized TiAu-contact metallization utilized as a topside-contact metal for GaAs SCOW devices. The sputter-deposition conditions and film-thickness control of individual metal films have been optimized to minimize film-stress and post-deposition wafer bow change as a function of time. Devices fabricated with this improved metallization process are blister-free, providing high device yields regardless of device-processing timing.

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CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

ELECTRONIC SUPPLEMENTARY MATERIAL

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