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Low-temperature joining of semiconductor substrates and thin layers on solid and flexible wafers by solid-liquid inter-diffusion bonding using a low-thickness Ni-Sn system

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Abstract—In this study, we investigate the conditions of bonding by implementing Transient Liquid Phase of the metal technique in order to secure a flexible polyimide sheet to different semiconductor substrates (Si, SOI, InP). We test the TLP bonding using Ni and Sn as filler and parental metals at low thicknesses (from 300 nm/600 nm to 1.5 μm /3 μm , for Ni/Sn respectively). At these conditions, we highlight a discrepancy with the expected behavior, especially above 280°C.

Index Terms—Packaging, Transient Liquid Phase, Ni-Sn, Low Thickness, Flexible

I. INTRODUCTION

Electronic devices operating in high-temperature environments are critical for a wide range of applications. However, packaging such devices is a major challenge in the fabrication process, especially when multiple welding steps are involved. The packaging must be able to withstand high temperatures and wide temperature cycling. In this process, it is required to avoid re-melting and to have low susceptibility to creep-fatigue failure, which becomes more critical at operating temperatures close to the melting point of the bonded material [1].

Transient liquid phase (TLP) bonding is an innovative method used to bond metallic or ceramic materials. It has become an attractive bonding technology in 3D chip stacking due

to its relatively low-temperature bonding and high-temperature service [2]. The formation of Intermetallic Compounds (IMCs) starts at the interlayer melting temperature, which is 232°C for widely implemented Sn-based soldering [3]. Over time, the liquid can fill any unevenness of the substrate, while the parental metal dissolves into the liquid, forming a homogeneous IMC layer.

Although IMCs usually exhibit good mechanical performance at both room and high temperatures, Sn-based joints are known to form voids during the bonding process. Extensive research has been done on the subject in the last decades, and the procedure to prevent voids due to volume shrinkage is well known [1, 4–6]. A minimum thickness of filler metal is required to avoid overconsumption due to parental metal interdiffusion. Despite applying the calculated critical thickness, we observed defects at low thicknesses, in contrast with the expected behavior. This suggests that a minimum amount of material is necessary for the process to work properly.

This study focuses on TLP bonding using Ni and Sn, as filler and parental metals (Ni-Sn-Ni). There are few detailed investigations on low-thickness, under a few μm , e-beam metalization bonding [7]. We implement this method by varying the thickness of the layers from 300 nm/600 nm

II. EXPERIMENTAL DETAILS

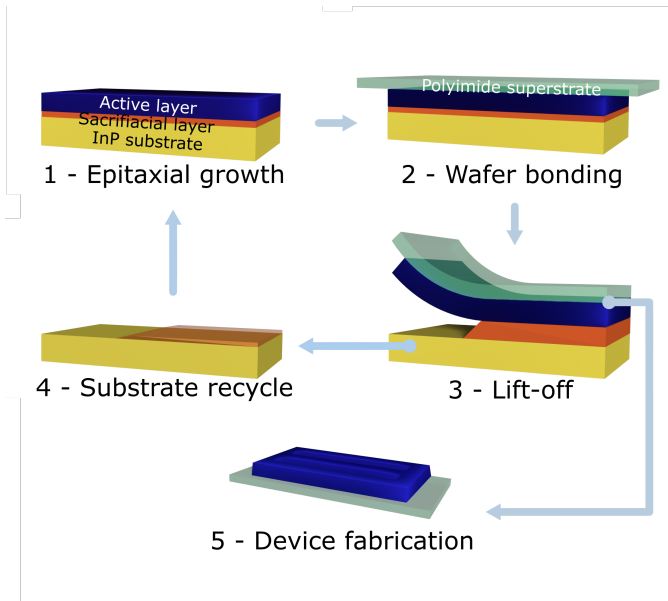


Fig. 1. Representation of the main steps of the Epitaxial lift-off process.

to 1.5 $\mu\text{m}/3 \mu\text{m}$, for Ni/Sn respectively. We study the alloy (Ni_3Sn_4) formation and how it is affected by the bonding process. We relate the bonding process parameters such as heating rate, holding time, temperature, and pressure, to the formation of the alloy, by testing the adherence strength and the presence of defects in the joint. Finally, we verify its application on different substrates, such as Si, SOI, and InP, joint to flexible polyimide sheets. Indeed, the future perspective of this work is to implement this method for other applications. In particular, we want to exploit its ability to bond rough surfaces, like polyimide, in the frame of Epitaxial Lift-Off (ELO), depicted in Fig.1, aimed to attach InGaAs layers, to be released and transported through from the native InP substrate to a flexible host holding [8].

With the future perspective of utilizing the transient liquid phase (TLP) bonding technique to attach an InP/InGaAs system onto a polyimide substrate, as a starting point, we employ a Si substrate that is both cost-effective and rigid. Afterward, we apply this process onto an SOI substrate, to test the feasibility of the Si thin film release and its transport onto the superstrate. Finally, we test the bonding on the proper substrate, InP bonded to the polyimide superstrate. For Si and SOI, the tested samples consisted of $\frac{1}{4}$ of a 4-inch wafers, corresponding to an approximate area of 19.6 cm^2 . In contrast, the InP samples utilized 2-inch wafers. All the substrates, Si, SOI, and InP, underwent identical preparation procedures, which included an acetone/isopropanol bath, followed by an O_2 plasma cleaning process. A metal deposition employing e-beam evaporation (by Intlvac evaporator) was performed, in order to obtain the following structure: as in Fig. 2, Pd (25 nm) as an adhesion layer, Au (150 nm), Ni as the parental metal, Sn as an intermediate layer, and Au (10 nm) as an anti-oxidation layer. The deposition process for the polyimide involved Cr (25 nm) as an adherence layer, Ni (300 nm) as the parental metal, and Au (25 nm) as an anti-oxidation layer. The superstrate was prepared in a facility where Sn deposition and Ni deposition thicker than 300 nm were not allowed. Our initial aim was to minimize the amount of deposited metal, which led us to select evaporation as the preferred deposition technique, because of its ability to provide precise control over the deposition rate, ensuring high uniformity and purity [9]. Moreover, given the complexity of the target structure (InP substrate - InAlAs/AlAs sacrificial layer - InGaAs active layer), it was important to perform this step at a low temperature to prevent possible stresses. Therefore, e-beam evaporation was considered the appropriate choice. The samples are subsequently bonded using an ALM Wafer Bonder, under the conditions shown in Tab.I.

TABLE I
BONDING CONDITIONS

Parameters	Values	
	Range	Best conditions
Heating rate	20°C/min, 30°C/min, 40°C/min	20°C/min
Holding time	20 min, 60 min, 120 min	120 min
Holding temperature	240°C, 260°C, 280°C	260 °C
Pressure	1 MPa	1 MPa
Ni/Sn Thickness	1.5 $\mu\text{m}/3 \mu\text{m}$ 900 nm/1800 nm 300 nm/600 nm	1.5 $\mu\text{m}/3 \mu\text{m}$

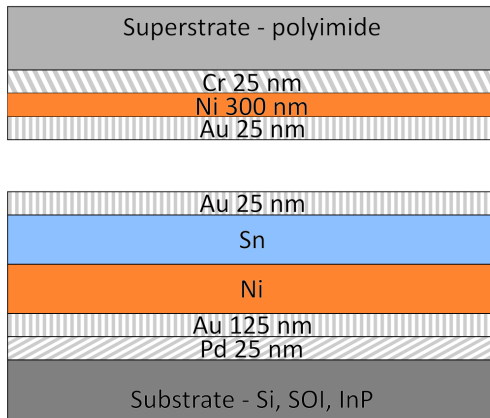


Fig. 2. Schematics of the structures

The primary requirements for our study are to ensure that the bonding does not induce stress in the released layer and that it withstands the subsequent etching step. Therefore, its quality has been assessed based on uniformity and adhesion strength. In terms of uniformity, any defects due to unevenness or un-bonded areas may lead to higher stress and detachment.

As for adhesion strength, the result of the experience with SOI lift-off served as a baseline for comparison.

As a result, we found that the best conditions were those that demonstrated the presence of a strong alloy formation, directly associated with bonding strength, while also exhibiting minimal defects such as detachments or overconsumption. Furthermore, the measurement of adhesion strength itself played a significant role in determining the optimal conditions. Moreover, in order to be compatible with ELO (Epitaxial Lift-Off) applications, it is essential that the bonding process does not result in the overflow of metal (liquid tin) onto the sidewalls. Such overflow would hinder the subsequent lift-off step, as it would prevent the sacrificial layer from being etched by HF (hydrofluoric acid) if it remains shielded by the metal.

The parameters values were chosen in order to satisfy the process requirements. In particular, holding time and temperature were strongly affected by the wafer bonder characteristics. Moreover, temperature has to be higher than Sn melting temperature (232°C) but lower than the polyimide working limit (namely 400°C, lowering in performance above 350°C [10]). The heating rate was chosen starting from examples found in literature [6]. The heating takes between 5 to 12 minutes; the cooling was determined by the wafer bonder and requires from 2 to 4 hours. Concerning pressure, the value of 1MPa comes from examples in literature [Bosc]. We attempted to lower the pressure to avoid overflow of metal on the sample's sidewalls, but this caused a decrease of adhesion and thus we kept the value at 1MPa.

To obtain a TLP bonding, the liquid phase of metal is necessary, in order to wet both parental metal surfaces. This is achieved by providing a sufficient amount of Sn, to avoid overconsumption during the formation of Ni_3Sn_4 . A model has been developed by Bosco et Zok [4] and Marauska et al. [11] for Cu-Sn, but it can be similarly applied to Ni-Sn. When this critical thickness is satisfied, based on the densities of the intermetallic compound, nickel, and tin, we can define a thickness for Ni which is half of the thickness for Sn. This will ensure the presence of a nickel layer at the end of the bonding process. This nickel layer will serve as a diffusion barrier between the semiconductor and the metals.

This sets a lower limit for Sn thickness, but the holding time is proportional to the thickness of the interlayer, meaning that the thicker the Sn layer the longer the process. The holding time was modified based on the IMC growth kinetics examined by [4]. The rate of Sn consumption, z_{Sn} , was evaluated as a function of temperature and time using the following equation [5]:

$$z_{\text{Sn}}(T, t) = \frac{\Phi}{\Phi + 1} \sqrt{k_0 e^{-Q/RT} t} = \frac{\Phi}{\Phi + 1} z_{\text{IMC}}(T, t) \quad (1)$$

with

$$\Phi = \frac{Y M_{\text{Sn}} / \rho_{\text{Sn}}}{X M_{\text{Ni}} / \rho_{\text{Ni}}} \quad (2)$$

where the transformation factor Φ characterizes the amount

of Sn consumed during the formation of an IMC layer for a specified thickness z_{IMC} , M represents the molar mass, ρ is the density of Sn and Ni, and $X = 3$ and $Y = 4$ as the stoichiometric ratios of the resulting IMC alloy, Ni_3Sn_4 .

III. RESULTS AND DISCUSSION

To achieve a homogeneous layer of alloy in the joint, the first step consisted in determining the best bonding conditions on the Si substrate. Then, we aimed to test the applicability of the process for ELO, wherein a thin layer grown on a substrate is selectively etched by removing a sacrificial layer. To assess its feasibility, we utilized an SOI substrate where the oxide layer served as the sacrificial layer and the Si membrane as the thin layer to transfer. This step is crucial to determine if the bond can survive the etching. Finally, the bonding process is applied to InP.

A. Si as substrate and bonding conditions evaluation

TLP bonding was performed initially with Ni-Sn-Sn-Ni, joining two Si substrates. To show the scallop-like structures, the process is halted before completion. EDS and XRD measurements indicate a higher presence of Sn in the central area where IMC formation is incomplete, meaning that the two fronts of formation did not reach each other.

After this step, the process is applied to bond the Si substrate to the polyimide superstrate. The bonding occurs with Ni-Sn-Ni, with a thickness of 1.5 μm /3 μm /300 nm respectively, and the process could be completed in less time than before, according to the expected holding time predicted by (1). At 240°C, the required holding time is 133 min. To understand the effects of time over the alloy formation, the process is repeated with holding times of 20 min, 60 min, and 120 min which, we expect, will still ensure a uniform joint. EDS

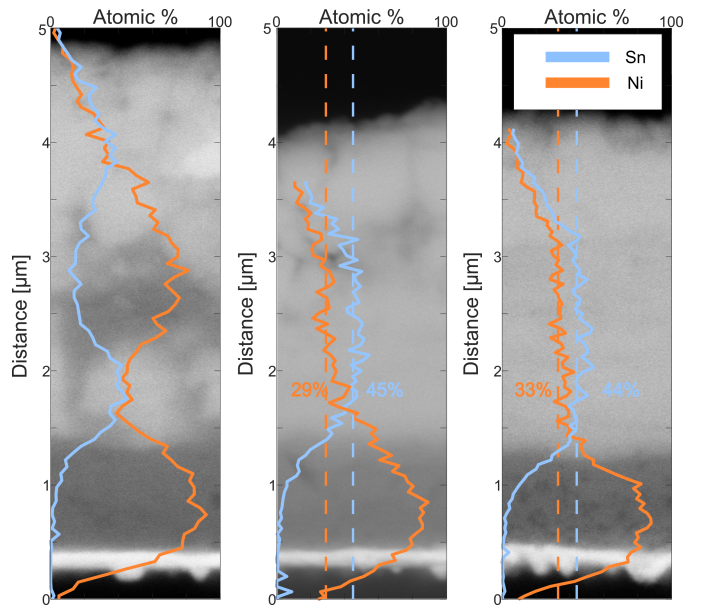


Fig. 3. EDS and SEM analysis of Si-on-polyimide superstrate bonding, varying holding time (20 min, 60 min, 120 min)

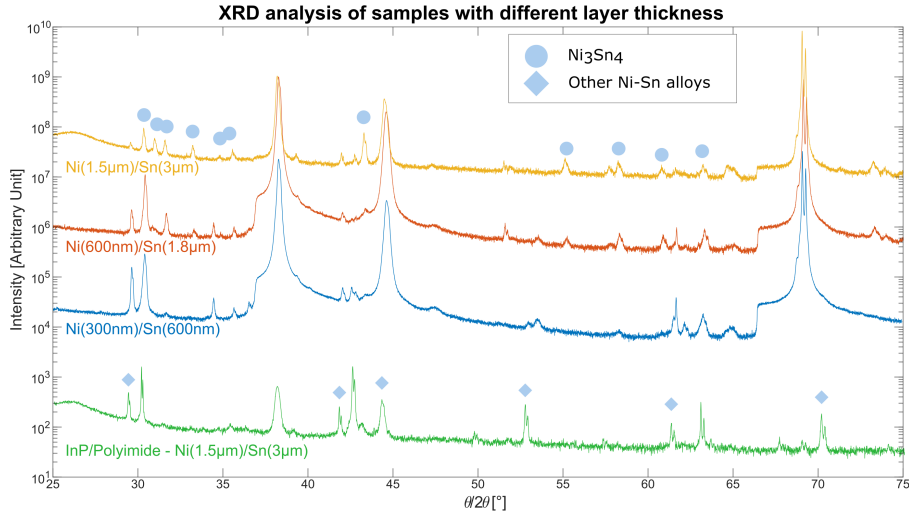


Fig. 4. XRD spectra of three Si/Polyimide samples, varying bonding thickness (yellow, red, blue) compared to InP/Polyimide spectrum (green). For clarity the peaks not related to the Ni_3Sn_4 have not been labeled.

measurements, shown in Fig.3, reveal the higher migration of Ni into Sn and an average ratio of the two closer to the expected one in Ni_3Sn_4 , meaning the total consumption of Sn. At this stage, it was necessary to address the issue of Sn overflow, which resulted in its leakage and deposition on the sidewalls. Therefore, the overflow was measured and a designated crown-shaped region was intentionally left free of metal during the deposition, to accommodate the inevitable overflow.

It is worth mentioning that the gold layer does not represent an obstacle to the migration of Ni from one half to the other. This has been verified first with EDS observations, where no relevant amount of gold was found. Additionally, a test structure where Ag replaced Ni just on one side, was used to further confirm that Ni, coming from the polyimide side, diffuses successfully on the other half and forms Ni_3Sn_4 alloy with Sn. Therefore, we can conclude that gold does not hinder the nickel diffusion process.

We test different temperatures (240°C, 260°C, 280°C), all applied during the same reference time of 20 min, for comparison. According to (1), the process should not be completed at any of the chosen temperatures. Nevertheless, at 280°C, defects related to the overconsumption of Sn in the form of holes in the alloy area and increased porosity in the pure Ni area are observed with SEM.

Upon decreasing the thickness of both Ni and Sn to 900 nm and 1800 nm respectively, the bonding displays comparable features on SEM and XRD observations, as illustrated in Fig.3. However, an adhesion test reveals weaker bonding in the samples. As for thinner layers of Ni/Sn (300 nm/600 nm), no improvement in bonding is observed even with an increase in time or temperature, in contrast with (1). In the previous case with Ni(900 nm)/Sn(1500 nm), although the bonding was

weaker due to lower alloy formation, Ni_3Sn_4 was detected on both halves, suggesting the transfer of Ni from both sides. In the Ni(300 nm)/Sn(600 nm) case, our findings show no signs of Ni_3Sn_4 alloy formation, as shown in Fig. 3, and no bonding occurs between the two halves. Indeed, TLP bonding requires the formation of a bulk liquid phase to create a consolidated bond [6]. Thus, we conclude that the thickness of the metals directly influences the quality of bonding, regardless of other parameters.

B. SOI as substrate and lift-off application

The bonding on SOI joined to a polyimide sheet is performed to verify that the process could be applied in the frame of epitaxial lift-off, consisting of etching in buffered HF (BOE) solution. The Ni/Sn metal layers have a thickness of 1.5 μm /3 μm and 900 nm/1800 nm. However, the second combination does not withstand the lift-off process and this option has been therefore discarded. For Ni(1.5 μm)/Sn(3 μm), the membrane of Si has been completely detached from the substrate and transported on the polyimide superstrate. This indicates that this thickness is appropriate to perform the bonding, thus we applied it in all subsequent samples.

C. InP as substrate

Finally, the bonding process is applied on an InP substrate with the metalization Ni(1.5 μm)/Sn(3 μm) validated before. We expected to find a better result in terms of adhesion and uniformity with InP, since the thermal expansion coefficients of Ni and Sn are closer than for Si. Nevertheless, the bonding is not uniform along the surface, revealing the presence of unbonded areas, between the polyimide and the substrate. After an XRD analysis, shown in Fig.3, we see that there is no trace of the Ni_3Sn_4 alloy, strongly related to the strength of the bonding. Since this case does not meet the expectations,

it requires further investigation, before proceeding with TLP bonding of an InP/InGaAs system on a polyimide superstrate.

IV. CONCLUSIONS

This work aims to explore the effects of TLP bonding, decreasing the thickness of the metal layer. We tested three systems, maintaining the relative ratio between Ni and Sn: 1.5 μm /3 μm , 900 nm/1800 nm, and 300 nm/600 nm. The bonding conditions have been optimized for the Si substrate and the lift-off process was tested on SOI. From this study, we present the following conclusions: (a) longer isothermal holding time, at 240°C, leads to a more complete the formation process for Ni₃Sn₄ alloy: in agreement with previous studies on the topic; (b) temperature increases led to defects due to the overconsumption of Sn, within a shorter holding time than expected; (c) moreover, despite varying temperatures and holding time, there is a minimum thickness that allows the bonding to survive the epitaxial lift-off process, which is close to Ni(900 nm)/Sn(1800 nm). Its application on InP is still under investigation.

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REFERENCES

- [1] O. Mokhtari. “A review: Formation of voids in solder joint during the transient liquid phase bonding process - Causes and solutions”. In: *Microelectronics Reliability* 98 (2019), pp. 95–105.
- [2] L. Zhang and S.J. Zhong. “Effect of Ni₃Sn₄ on the Thermo-Mechanical Fatigue Life of Solder Joints in 3D IC”. In: *Frontiers in Materials* 8 (2021).
- [3] J. Wang et al. “Transient liquid phase bonding of Sn-Pb solder with added Ni particles”. In: *2022 23rd International Conference on Electronic Packaging Technology (ICEPT)*. IEEE, 2022, pp. 1–6. ISBN: 978-1-6654-9905-7.
- [4] N. S. Bosco and F. W. Zok. “Critical interlayer thickness for transient liquid phase bonding in the Cu–Sn system”. In: *Acta Materialia* 52.10 (2004), pp. 2965–2972.
- [5] A. Lis and C. Leinenbach. “Effect of Process and Service Conditions on TLP-Bonded Components with (Ag,Ni–)Sn Interlayer Combinations”. In: *Journal of Electronic Materials* 44.11 (2015), pp. 4576–4588.
- [6] G. O. Cook and C. D. Sorensen. “Overview of transient liquid phase and partial transient liquid phase bonding”. In: *Journal of Materials Science* 46.16 (2011), pp. 5305–5323.
- [7] J. Cao et al. “A low-temperature sealing method for metal-supported oxide fuel cell applications: Ni–Sn transient liquid phase bonding”. In: *Vacuum* 187 (2021), p. 110048.
- [8] F. Chancerel et al. “Epitaxial lift-off of InGaAs solar cells from InP substrate using a strained AlAs/InAlAs superlattice as a novel sacrificial layer”. In: *Solar Energy Materials and Solar Cells* 195 (2019), pp. 204–212.
- [9] S. Lemettre et al. “Low temperature solid–liquid interdiffusion wafer and die bonding based on PVD thin Sn/Cu films”. In: *Microsystem Technologies* 23.9 (2017), pp. 3893–3899.
- [10] C. Yi et al. “High-temperature-resistant and colorless polyimide: Preparations, properties, and applications”. In: *Solar Energy* 195 (2020), pp. 340–354.
- [11] S. Marauska et al. “Low temperature transient liquid phase bonding of Au/Sn and Cu/Sn electroplated material systems for MEMS wafer-level packaging”. In: *Microsystem Technologies* 19.8 (2013), pp. 1119–1130.