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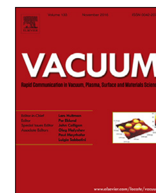

Diffusion brazing of monocrystal silicon by using germanium as filler material

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ABSTRACT

In this research, diffusion brazing of monocrystal silicon was performed using germanium powders as interlayer. The effects of brazing temperature (950–1050 °C) and time (1–3 h) were studied on the joint microstructure and its mechanical properties. The results indicated that, with the rise in brazing time and temperature, the morphology near the sample edge turned from sintered structures to bridge connections. While those in the center evolved from straight Ge layers to symmetric layer structures with the sequence of "Si base/Isothermal solidification zone/Athermal solidification zone/Ge layer". Withal, the crushing and interlocking morphologies appeared. Bond strength was evaluated and maximum tensile strength over 15.8 MPa was obtained for the joint processed at 1000 °C for 3 h. Results showed that, thickness of diffusion layer (i.e., the isothermal solidification zone and athermal solidification zone) was proved to be the controlling factor for the tensile strength of diffusion brazed monocrystal silicon.

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1. Introduction

Monocrystal silicon, a semiconductor material, is widely used in the fields of electronic packaging, micro electro mechanical system (MEMS), and optoelectronic devices [1,2]. However, monocrystal silicon is brittle and difficult for machining, thus a great challenge is aroused for its bonding technique. Conventionally, monocrystal silicon bonding is limited to wafer bonding [3–5]. Direct bonding is a normal way, which is a solid diffusion method to bond silicon wafer in micrometer size with, however, low bond strength. Because of the requirement of gas tightness and bonding strength, direct bonding is unsuitable for bulk material, where large mating surface increases difficulty for homogeneous interface diffusion. For bonding of bulk monocrystal silicon, B–B Sun et al. [6] introduced Au–Si eutectic method, in which liquid was formed that could be able to wet the interface of the base materials. However, it's inevitable that voids have been found in the joint resulted from no intermiscibility of Au–Si system at low temperature, and solidification contraction and intrinsic difference [6–8]. Thus, it is reasonable to overcome such defects by selecting silicon related binary isomorphous system, where germanium comes into being. Si–Ge forms solid-solution with mutual solubility in all composition range. Besides, the SiGe semiconductor alloy prepared by RF sputtering [9], chemical vapor deposition [10] or ion implantation [11] has gained considerable attention due to its potential applications in heterojunction bipolar transistors and high electron mobility field effect transistors. However, there is limited published information in the literature regarding the bonding of bulk monocrystal silicon with Ge interlayer. Yet, K.Y. Byun et al. [12,13] and H. Kanbe et al. [14] have achieved Ge/Si direct wafer bonding, which is a forceful basis for experimental study of Ge/Si brazing. Ge and Si are the same main group elements, which have many similar properties and the same lattice structure with only 4.2% lattice mismatch [15]. Consequently, it is theoretically feasible to achieve solid solution joint of monocrystal silicon with Ge interlayer.

In this work, efforts were made to produce the diffusion brazed joints of monocrystal silicon using Ge powders as interlayer and with a focus on formation of various interface microstructures at different temperatures for different brazing time and their effects on mechanical properties.

2. Experimental set up and methodology

The base material is monocrystal silicon with 99.9% purity, supplied by Shaanxi Huashan Semiconductor Material Factory, and

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pure Ge powders (microns) with 99.999% purity, supplied by Chinese Medicine Chemical Reagent Co. Ltd, was employed as interlayer material. The samples were cut into ϕ30 mm × 4 mm with crystal plane (100) to be the mating face, which were polished (Ra = 0.03 μm), supersonically cleaned in acetone and dried by cold air. The powders (2 g) were dispersed in solvent of 20 ml ethyl alcohol with 3 ml polyethylene glycol by stirring in electromagnetic agitator for 2 h. Thereafter, the dispersion was brushed onto the silicon surface and then air dried, the process of which was repeated 5 times till the powders had covered the surface (~10 μm thick). Diffusion brazing experiments were conducted at three different temperatures of 950, 1000 and 1050 °C (i.e., above the melting point of Ge: 938 °C) for 2 h to study the temperature effect, while the brazing time of 1 h and 3 h were added at 1000 °C to study the effect of holding time. The samples were mated in sandwich structure by two silicon samples, of which one had been covered by Ge layer, and then loaded into vacuum radiation heating furnace (FJK-2, Northwestern Polytechnical University) between the two extrusion heads, as shown in Fig. 1. The extrusion head was used to apply an extrusion force (hydraulic-drive) onto the sample, i.e., welding load. The masks (mica foils) were used to prevent sample and head from adhering together. The samples were heated up to the brazing temperature with a heating rate of 10 °C/min. After brazing, the samples were cooled down in furnace. The load of 1.2 MPa was applied along the longitudinal direction of the samples in 2×10^{-3} Pa vacuum, under which the excessive Ge melts had been squeezed out of the interface so that leave the same residuals for bonding.

Once the brazing process was completed, the brazed samples were cut transversely through the bond using a wire cut electric discharge machine for metallographic examination. The cross-sections of these joints were metallographically polished for microstructure analysis by Optical Microscope (OM, OLYMPUS-GX71), Scanning Electron Microscope (SEM, JSM-6390) and Energy Dispersive Spectrometer (EDS). Additionally, a sample size of 10 mm × 10 mm × 8 mm was prepared for tensile test (Universal tensile testing machine, Instron 4505), both sides of which were adhered to fixtures by AB resin glue for holding of the sample because the silicon was too crisp to be clamped during the test. In addition, the tensile strength of the fixtures sticked by AB resin glue without samples had been tested for 15.8 MPa, i.e., the binding force of AB resin glue. As a consequence, if the samples fractured on the AB resin glue, their strength over 15.8 MPa was proved, otherwise, less than 15.8 MPa.

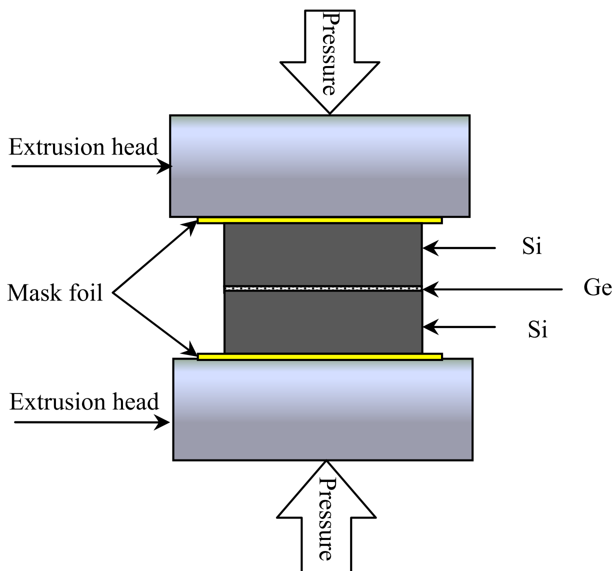

Fig. 1. Experiment setup showing assemble for diffusion brazing Si with Ge interlayer.

3. Results and discussion

3.1. Sintered interfacial morphology

Fig. 2 showed the interfacial morphologies occurred at the sample edge where there were found the sintered structures formed by Ge powders. According to the engineering method by Wengke soff, the normal pressure on the interface decreases along the radial direction when a column model is compressed by an axial force [16]. The sintered structures were caused by loose contact between particles not been pressed tightly adjacent to the edge. Fig. 2a showed the joint brazed at 950 °C for 2 h, where the particles' profiles remained. There were vertical cracks across the layer indicating the volume shrinkage during cooling as Ge layer remained porous. Though, melting of Ge powders was inadequate, a white line formed at Si surface implied the apparent diffusion (2 h of brazing time).

Fig. 2b showed the joint brazed at 1000 °C for 1 h, i.e., the brazing temperature was increased by 50 °C. The more continuous interfacial structure was found with sintered particles (gray) around the solidifying bulk crystals (white). This phenomenon indicated nearly complete melting of Ge powders. Craters dissolved and filled by Ge were formed at Si surface indicating strong Ge/Si interaction at this temperature. Such results implied possible brazing temperature (around 1000 °C) necessary for the melting of Ge powders, which corrected the effect of uneven distribution of pressure.

.2. The dissolution craters morphology

Fig. 3 showed dissolution craters morphologies found at the interface of joints brazed under different experimental conditions, where Fig. 3a and b illustrated the joints at 950 °C and 1050 °C for 2 h, and Fig. 3c and d at 1000 °C for 1 and 3 h. These results were consistent with the study by P.M. Zavracky et al. [17] and by Tabata. O et al. [18] where Si (100) plane served as the mating face, once the temperature reached the melting point of Ge, other crystal faces would be prior dissolved by Ge melts the lowest surface energy and slowest corrosion rate of Si (111), leaving inverted island structures with (111) plane as the boundary. The triangle and island craters in different shapes were consistent in essence, but observed in two parallel cross sections.

When the temperature was increased to 1050 °C, the quantity and size of craters increased and the interface of Ge/Si became curved. But the sawtooth configuration in the 1000°C/3 h joint implied that, the brazing time took more dramatical effects on the dissolution behavior than the temperature, as shown in Fig. 3c and d.

Considering the distribution of pressure along the radial direction, Ge craters mainly formed in the center of 950°C/2 h and 1000°C/1 h joints. Increasing temperature and brazing time corrected the effect of uneven distribution of pressure, thus craters were located along the whole interface.

3.3. The symmetric layer structure

Fig. 4 presented the symmetry microstructure of joint brazed at 1000 °C and holding for 3 h, it was observed by SEM in the secondary electron mode. It was viewed at the sample center where Ge melt fully but not easy to extrude out as far away from the edge, so it had a larger thickness and can be described as thick welding areas

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