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Influence of Ga Content on the Microstructure and Mechanical Properties of Cadmium-Free Filler Metal

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Abstract: The influence of Ga content on the melting temperature, wettability, microstructure, and mechanical properties of low-silver 12AgCuZnSn-2In-0.15Pr cadmium-free filler metal was investigated systematically by means of differential thermal analysis, X-ray diffractometer, scanning electron microscopy, energy-dispersive spectrometer, etc. The results showed that the addition of the Ga element reduced the solidus and liquidus temperatures of the novel low-silver filler metals, and effectively increased the spreading area of the filler metal on the copper and 304 stainless steel substrates. Furthermore, an appropriate amount of Ga element significantly optimized the interface morphology and improved the mechanical properties of the brazed joints. When the Ga content was 1wt.%, the shear strength of the brazed joints reached a peak value of 448 MPa, and the corresponding fracture morphology showed typical ductile characteristics with obvious dimples.

Keywords: low-silver filler metals; melting temperature; wettability; microstructure; shear strength

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1. Introduction

In the silver-based filler metals family, AgCuZnCd series alloys have been widely used in major projects and daily life related to the national economy and people's livelihood for their excellent brazing performance, and are known as "industrial universal glue" [1,2]. In recent years, with the rapid development of science and technology as well as the increasing awareness of environmental protection, many countries have implemented a series of regulations such as restriction of hazardous substances (RoHS) and the waste electrical and electronic equipment (WEEE) to limit the use of AgCuZnCd brazing filler metals in electrical and electronic equipment [3,4]. Among a number of cadmium-free silver filler metals, AgCuZnSn filler metals with silver contents of 34–56% are considered promising alternatives to AgCuZnCd filler metals [5]. However, metallic silver has dual use as an industrial product and financial product, and its high price limits the further use of high-silver filler metals and promotes the development of low-silver filler metals [6]. The Ag content in AgCuZnSn filler metal is lowered down, even to 20 wt.%, which, however, triggers several other issues, especially higher melting temperature and poor wettability, and thus distinctly deteriorates the brazing performance of the filler metal [7,8]. Therefore, it is imperative to develop and research novel high-performance, low-silver, and cadmium-free silver filler metals.

Thus far, the major approach for enhancing the performance of the low-silver filler metal is alloying [9], effectively via various elements, such as Sn [10], In [11], Mn [12], Li [13], and rare earth elements [14], contributing to a pronounced optimization in wettability and mechanical properties. In a previous study, In and Pr were selected to modify the brazing performance of 12AgCuZnSn filler metal [15], when the contents of In and Pr were 2 wt.% and 0.15 wt.%, the solidus temperature and liquidus temperature of the low-silver filler metal decreased to 756 °C and 782 °C, respectively, meanwhile, the wettability, microstructure, and mechanical properties of 12AgCuZnSn filler metal were also remarkably

improved. In order to further optimize the melting characteristic and mechanical properties of the low-silver filler metal, this paper selects the Ga element to modify the 12AgCuZnSn-2In-0.15Pr filler metal. Metal Ga is half the price of metal Ag, and the melting and boiling points of Ga are 29.8 °C and 2403 °C, respectively. The Ag-Ga and Cu-Ga [16] binary phase diagrams are shown in Figure 1, as we can see from the figures that the solid solubilities of Ga in Ag and Cu are large, meanwhile, the melting temperatures of copper and silver alloys decrease with the addition of Ga, which is beneficial to the low-silver filler metal.

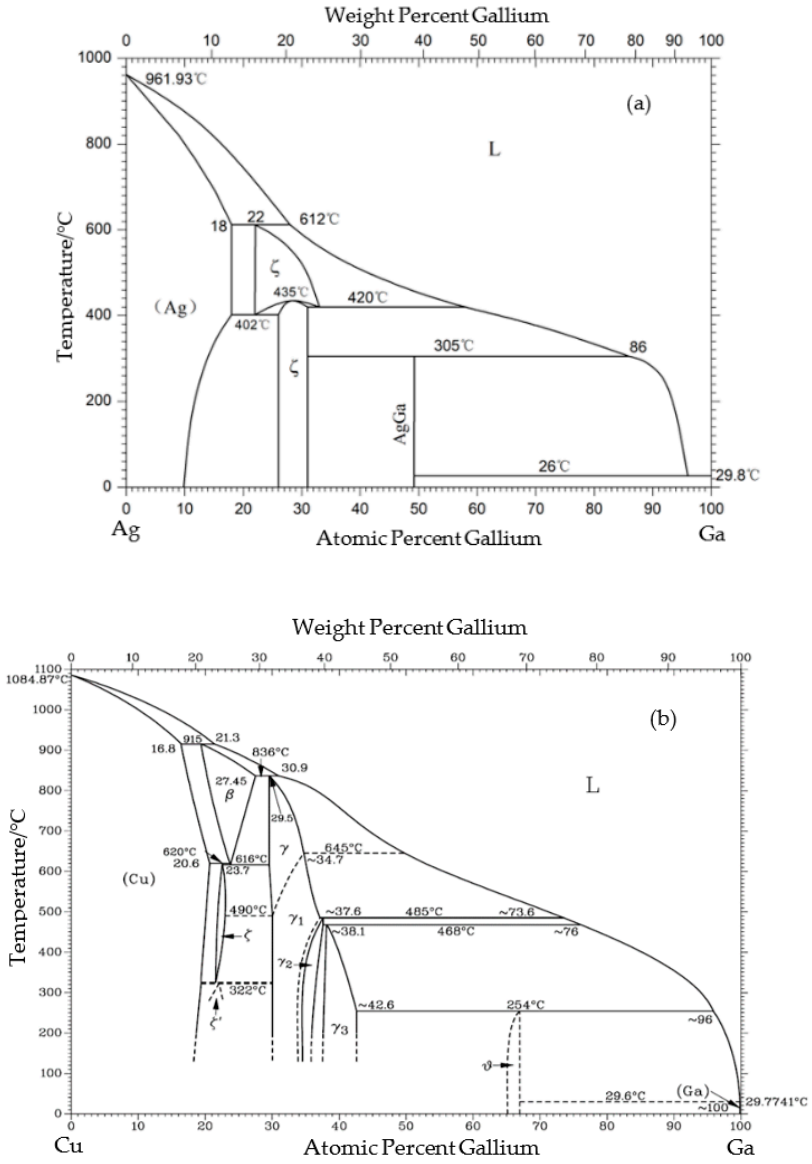

Figure 1. Ag-Ga and Cu-Ga binary phase diagrams: (**a**) Ag-Ga binary phase diagram, (**b**) Cu-Ga binary phase diagram [16].

In the present investigation, the influence of Ga addition on the microstructure and properties of 12AgCuZnSn-2In-0.15Pr low-silver filler metal for brazing copper and 304 stainless steel was studied. The wettability of the filler metals with designed additions of Ga on the copper and stainless steel substrates and the fracture morphologies of the brazed joints were investigated.

2. Materials and Methods

Pure Ag, Cu, Zn, Sn, In, Ga (99.9 wt.% purity), and Cu-10Pr master alloy were used as raw materials and melted in a medium frequency furnace (frequency 600 Hz, power

110 kW). The molten alloy was held for about 15 min, with repeated mechanical stirring so as to ensure homogeneity. The melted alloys were cast into ingots in 50 mm diameter steel molds and then cut into alloy bars about 80 mm in length. All the alloy bars were drawn into a wire with a 1.9 mm diameter for brazing. The chemical compositions of the novel low-silver filler metals were investigated using an inductively coupled plasma atomic emission spectrometer (ICP-AES), the results are listed in Table 1.

Table 1. Chemical composition of the novel low-silver filler metals (wt.%).

No.	Ag	Cu	Zn	Sn	In	Pr	Ga
1	12.0	Bal.	37.8	1.5	2	0.15	0
2	12.0	Bal.	37.5	1.5	2	0.15	0.5
3	12.0	Bal.	37.2	1.5	2	0.15	1
4	12.0	Bal.	36.7	1.5	2	0.15	2
5	12.0	Bal.	36.1	1.5	2	0.15	3
6	12.0	Bal.	35.0	1.5	2	0.15	5

The solidus and liquidus temperatures of the filler metals were determined using differential thermal analysis (DTA, HCR-1, HENVEN, Beijing, China), and the heating was done at 10 °C/min from room temperature to 900 °C in an N_2 gas flow. The copper and 304 stainless steel were processed into plates with dimensions of 40 mm × 40 mm × 2 mm for the spreading test and 60 mm × 25 mm × 3 mm for the shear strength test, the schematic illustrations are shown in Figure 2. All filler metals and specimen surfaces were polished with SiC papers to remove the oxide film, and then ultrasonically cleaned with ethanol. The spreading test was performed according to China's National Standard GB/T 11364-2008 [17], the filler metals (0.2 g) were placed in the center of the specimens, and FB102 flux (40% of KF, 25% of KBF_4, 35% of B_2O_3) was applied, which was heated at 850 °C for 1 min in an electrical resistance furnace. The spreading area was measured using the software Image-Pro Plus (Media Cybernetics, Rockville, MD, USA).

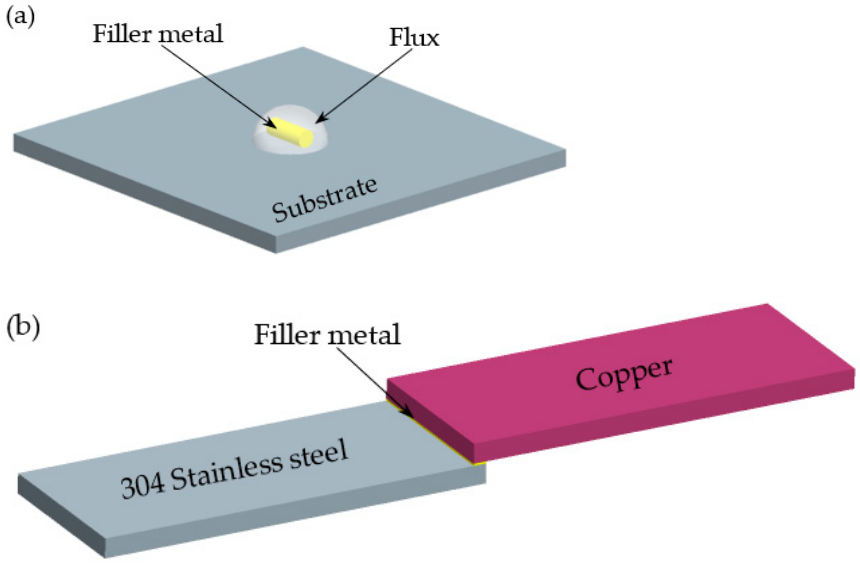

Figure 2. (**a**) Schematic illustration of the spreading specimen; (**b**) schematic illustration of the brazing specimen.

The flame brazing method was used in the present work for brazing copper and 304 stainless steel, and the shear strength of brazed joints with an overlap length of 2 mm was tested using an electronic universal testing machine, and the constant loading rate of 5 mm/min at room temperature was applied in this study. The shear test was conducted based on China's National Standard GB/T 11363-2008 [18].

The novel filler metals and the cross section of brazed joints were prepared by standard polishing techniques and etched with a solution of $(NH_4)_2S_2O_8$ (15 g) + H_2O (100 mL) + NH3·H2O (2 mL), and then, the mounted specimens and fracture morphologies of brazed joints were characterized employing scanning electron microscopy (SEM, ZEISS S IGMA 500, Oberkochen, Germany) and an energy-dispersive spectrometer (EDS, Bruker Nano XF Lash Detector 5010, Billerica, MA, USA).

3. Results

3.1. Melting Temperature and Wettability of the Filler Metals

The solidus and liquidus temperatures of 12AgCuZnSn-2In-0.15Pr-*x*Ga filler metals obtained from DTA analysis are shown in Figure 3. It can be seen that the addition of a trace amount of Ga can effectively reduce the melting temperatures of the filler metal. When the addition of Ga reaches 2 wt.%, the solidus and liquidus temperatures of the filler metal decrease to 719 °C and 761 °C, respectively. With the further increase of Ga content, the downward trend of the solidus and liquidus temperatures gradually flattens out. Ma et al. [19] found that the decrease of both solidus and liquidus temperatures is due to the low melting point of Ga, and appropriate amounts of Ga could dissolve in silver and copper matrix to form silver-based and copper-based solid solutions with lower melting points.

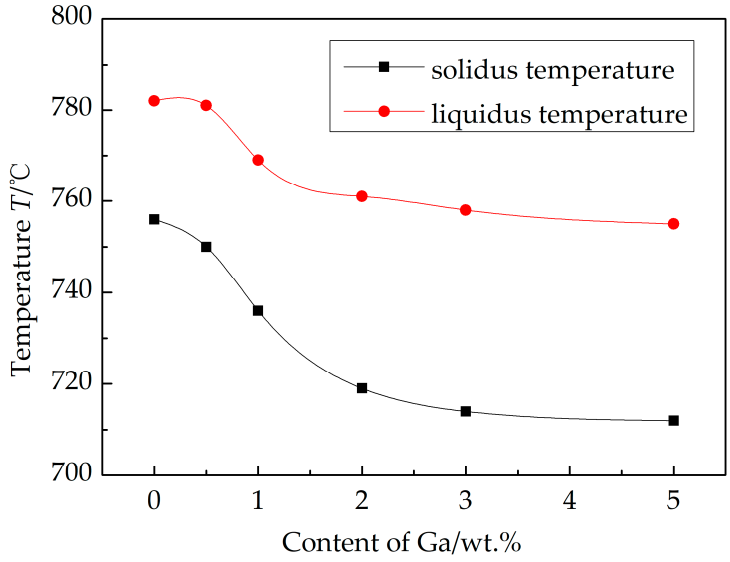

Figure 3. Melting behavior of 12AgCuZnSn-2In-0.15Pr-*x*Ga filler metals.

The wettability of filler metals is an extremely important indicator that determines the mechanical properties and reliability of brazed joints. In this research, wettability is assessed by the spreading area of the filler metals on the substrate, and the greater the spreading area, the better the wettability [20]. Figure 4 shows the spreading test results of the novel low-silver filler metals on copper and 304 stainless steel plates at the temperature of 850 °C. As shown in the figure, with the addition of Ga, the spreading area of the filler metals is significantly increased, when the Ga content is 2 wt.%, the spreading areas of the filler metals on copper and 304 stainless steel are 409 mm^2 and 354 mm^2, respectively. However, as the Ga content continues to increase, the increasing trend of the spreading area tends to gradually become more gentle. Therefore, combining the results of Figures 3 and 4, the improvement in the spreading performance of the novel low-silver metals may be mainly attributed to the decrease in melting temperature. Under the same brazing process parameters, the lower the melting temperature of the filler metal, the smaller the attraction of the atoms inside the liquid alloy to the surface atoms, that is, the smaller the interfacial tension, the larger the spreading area of the filler metal on the substrate [21].

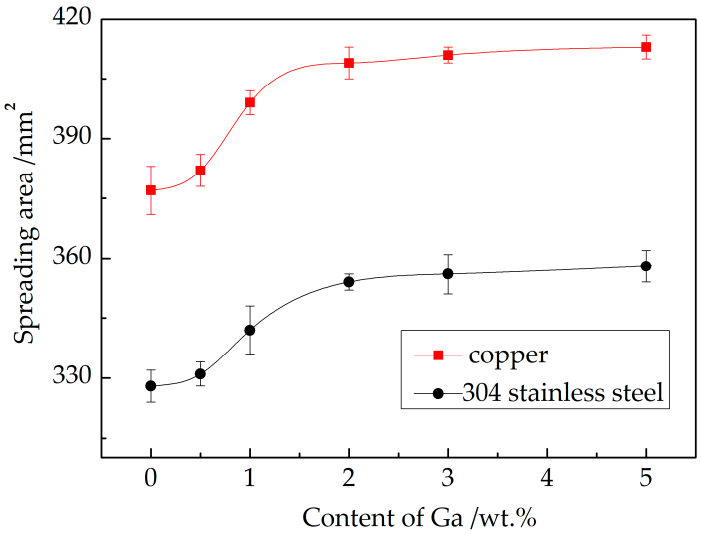

Figure 4. Spreading areas of the novel low-silver filler metals on copper and 304 stainless steel.

Figure 5 shows the SEM image and the EDX element mappings of the wetting boundary after the spreading test on 304 stainless steel using 12AgCuZnSn-2In-0.15Pr-2Ga filler metal. As can be seen from Figure 5, there is a "wetting ring" on the leading edge of the brazing alloy, which is beneficial to the wetting process between the filler metal and base metal [22]. Moreover, it can be seen obviously that the element mappings of Ag, In, and Sn overlap with the white reticular phase (wetting ring), and Fe, Cr, and Ni elements correspond to the gray blocky phase (304 stainless steel base metal). The preferential spreading of Ag-In-Sn liquid alloy can reduce the surface tension between the filler metal and 304 stainless steel substrate, and extremely improve the wettability of the filler metal [23]. However, it is noteworthy that there is no enrichment of Ga in the "wetting ring", which indicates that the influence mechanism of Ga on the wettability of the filler metal is different from that of In, and Ga tends to dissolve into Cu to form Cu-based solid solution with a low melting point, which improves the wettability of the filler metal on stainless steel base metal [24].

3.2. Microstructure of the Filler Metals

Figure 6 shows the X-ray diffraction patterns of 12AgCuZnSn-2In-0.15Pr-*x*Ga filler metals. As we can see from the XRD analysis results, the novel low-silver filler metals are mainly composed of an Ag-based solid solution, Cu-based solid solution, and CuZn compound phase (β-CuZn and a few brittle γ-Cu_5Zn_8). When the Ga content is 3 wt.%, the diffraction peaks of the γ-Cu_5Zn_8 phase increase, which is detrimental to the mechanical properties of the filler metals, therefore, the Ga content in the novel low-silver filler metal should be closely monitored. Notably, the new diffraction peak of the Ga phase arises when the Ga content reaches 5 wt.%.

Figure 7 shows the SEM images of the novel low-silver filler metals with different contents of Ga. It can be seen obviously that the addition of Ga to the 12AgCuZnSn-2In-0.15Pr filler alloy significantly improves its microstructure. When the Ga content is less than 2 wt.%, the microstructure of the filler metal is a network structure. Figure 8 shows the elements mappings of 12AgCuZnSn-2In-0.15Pr-2Ga filler metal in Figure 7e, the result indicated that the white needle-like phase overlaps with the element mapping of Ag, and the Ga element is uniformly distributed in the filler metal matrix. However, as the Ga content continues to increase, some gray phases (named as B region) and bright phases (named the C region) are formed in the matrix, the results of EDS shown in Table 2 indicated that the A and C regions are Ag-rich phases, in which the content of In is much higher than that of Ga, B, and D regions are Cu-rich phases that containing more Ga than In. Therefore,

it can be concluded that the Ga element prefers to form a solid solution with Cu, and In element tends to dissolve into Ag.

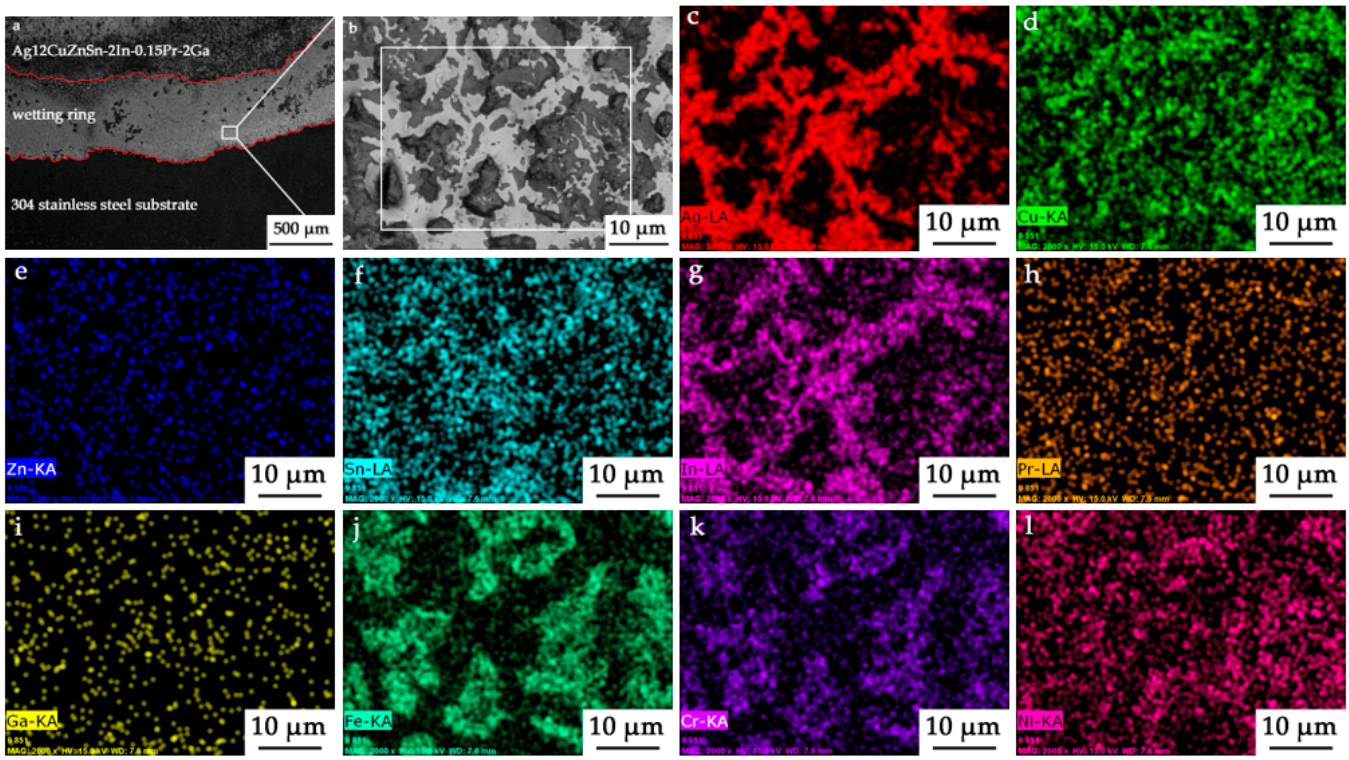

Figure 5. (**a**) SEM image of surface appearance after spreading of 12AgCuZnSn-2In-0.15Pr-2Ga filler metal on 304 stainless steel substrate, (**b**) high magnification SEM image of area marked with white squares in (**a**), (**c**–**l**) EDX element mappings of the distribution of Ag, Cu, Zn, Sn, In, Pr, Ga, Fe, Cr, and Ni, respectively.

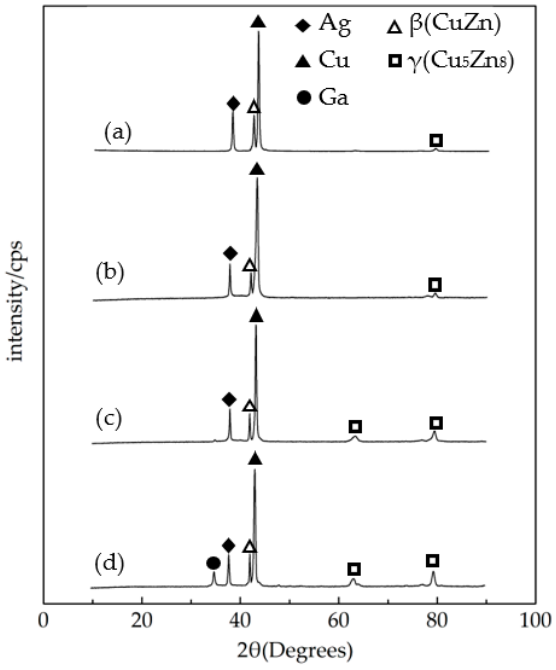

Figure 6. XRD patterns of the novel filler metals: (**a**) 12AgCuZnSn-2In-0.15Pr, (**b**) 12AgCuZnSn-2In-0.15Pr-1Ga, (**c**) 12AgCuZnSn-2In-0.15Pr-3Ga, and (**d**) 12AgCuZnSn-2In-0.15Pr-5Ga.

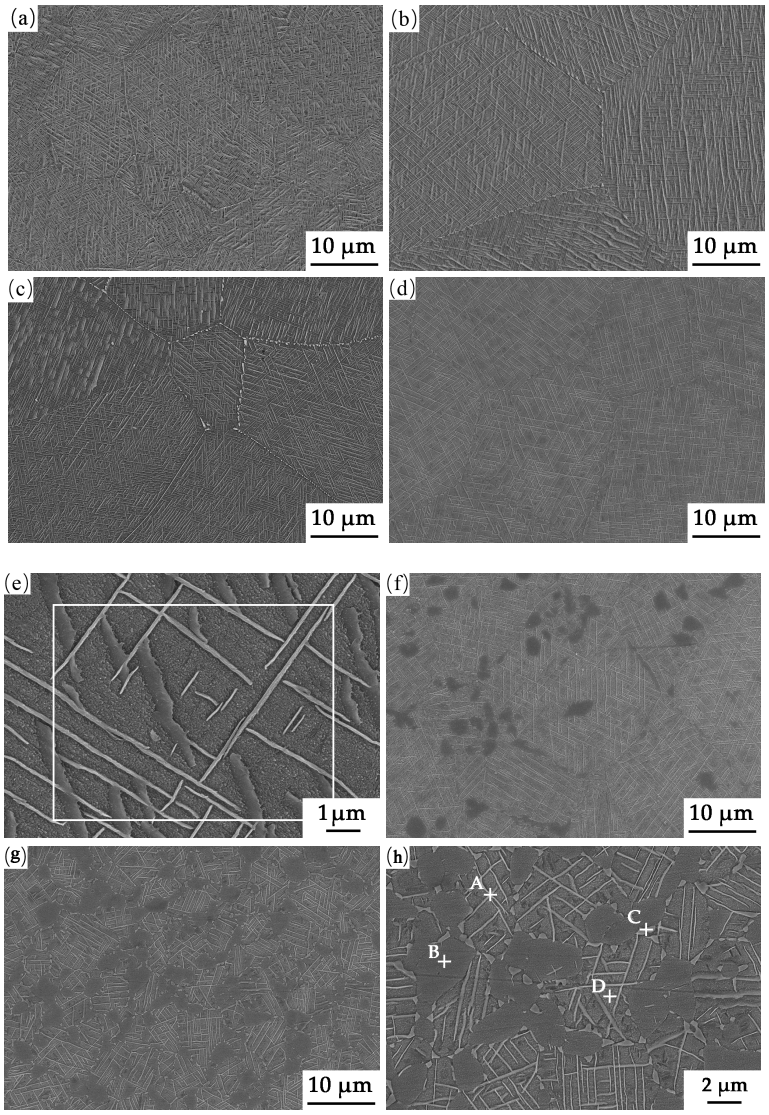

Figure 7. SEM images of microstructures of 12AgCuZnSn-2In-0.15Pr-*x*Ga filler metals ("*x*": the mass fraction of Ga): (**a**) *x* = 0, (**b**) *x* = 0.5, (**c**) *x* = 1, (**d**) *x* = 2, (**e**) *x* = 2 (high magnification), (**f**) *x* = 3, (**g**) *x* = 5, and (**h**) *x* = 5 (high magnification).

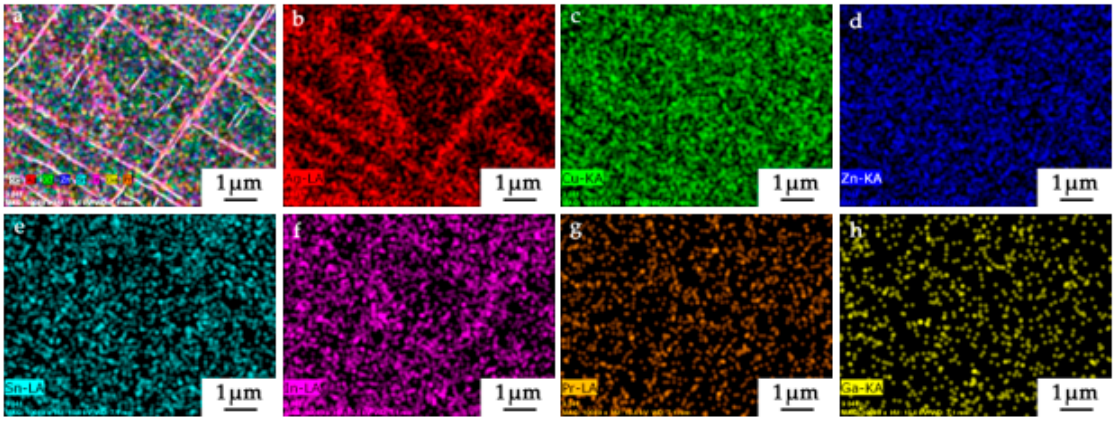

Figure 8. (**a**) The elements mapping of SEM image of 12AgCuZnSn-2In-0.15Pr-2Ga filler metal marked with white square in Figure 7 (**e**), (**b**–**h**) EDX element mappings of the distribution of Ag, Cu, Zn, Sn, In, Pr, and Ga, respectively.

Table 2. EDS analysis of the points indicated in Figure 7 (at.%).

Points	Ag	Cu	Zn	Sn	In	Pr	Ga
A	41.62	28.31	24.34	0.89	3.84	0.07	0.93
B	7.69	49.28	33.54	1.72	1.83	0.08	5.86
C	42.49	26.33	24.10	0.77	5.66	0.05	0.60
D	6.73	54.54	35.80	0.17	0.41	0.04	2.31

3.3. Microstructure and Mechanical Property of the Brazed Joints

The structure of the interface layer between the brazed seam and base metal has a significant effect on the mechanical properties of brazed joints [25]. Figure 9 shows the microstructure around the interface between the brazed seam and 304 stainless steel, and Table 3 shows the EDS analysis of the points indicated in Figure 9. As can be seen, the brazed seam is mainly composed of a white needle-like phase and a gray bulk phase. With the addition of Ga, the gray bulk phase begins to increase, meanwhile, the needle-like phase gradually transforms into the lamellar structure. Figure 9e and Table 3 show that the contents of Ag and In in the lamellar structure (named the B region) are higher than the gray bulk phase (named the D region), which indicates that the B and D regions are composed of an Ag-rich phase and Cu-rich phase, respectively. Notably, the content of Ga in the reaction layer (named the A region) formed at the interface between the brazed seam and 304 stainless steel substrate is higher than that of Ag, Cu, Zn, etc., indicating that Ga has a higher diffusion coefficient in 304 stainless steel. However, as shown in Figure 9h, when the Ga content reaches 5 wt.%, some bright Ag-In phases are formed in the brazed seam, which may deteriorate the mechanical properties of the brazed joint.

The shear tests of copper/304 stainless steel and 304 stainless steel/304 stainless steel brazed joints using 12AgCuZnSn-2In-0.15Pr-*x*Ga were performed at room temperature with a constant loading rate of 5 mm/min, and the test results showed that the fracture position of the copper/304 stainless steel brazed joints all occurred on the copper plate, the main reason is that the copper substrate is weak in strength and softened by heat annealing during the brazing process, indicating that the strength of the brazed seam is higher than that of the copper plate. Similar results were reported by Kuryntsev when laser welding of austenitic steel and copper was investigated, the fracture of the butt joint was also observed in the weakened copper heat affected zone metal [26,27].

The strength of 304 stainless steel is much greater than that of copper, and the fracture occurs in the interface layer between the brazed seam and 304 stainless steel in all cases during shear tests. The effect of Ga addition on the shear strength of the brazed joints is shown in Figure 10. As can be seen, when the content of Ga increased from 0 to 1 wt.%, the shear strength of the brazed joint increased gradually, and the peak shear strength reaches 448 MPa. The increase in shear strength of the brazed joint may be related to the microstructure of the filler metal after the addition of an appropriate Ga element, as shown in the Cu-Ga phase diagram where Ga could dissolve into Cu to form a Cu-based solid solution, in which the solid solution strengthening may be mainly responsible for the increase of shear strength. However, the shear strength begins to decrease with the further increase of Ga content. Moreover, when Ga content exceeds 3 wt.%, the brazed joint has an even lower shear strength compared to that of the Ga-free one.

The fracture morphology of the brazed joints can also explain the effect of Ga addition on the mechanical properties, as shown in Figure 11. With the addition of Ga in the filler metals, the fracture morphology of the brazed joints changed significantly. For the 12AgCuZnSn-2In-0.15Pr-1Ga brazed joint, the fracture surface exhibits a much finer and uniform dimple structure than that of the Ga-free one, as shown in Figure 11c, and the mechanical property of the corresponding brazed joint reaches the peak. However, with the increase of Ga content, the fracture morphology of the brazed joint begins to appear with secondary cracks and cleavage steps, which indicates that the fracture type of the brazed joint gradually changed from ductile fracture to cleavage fracture, and the mechanical

properties of brazed joints deteriorate significantly. Therefore, the content of Ga in the novel 12AgCuZnSn-2In-0.15Pr filler metal should be controlled within 2 wt.%.

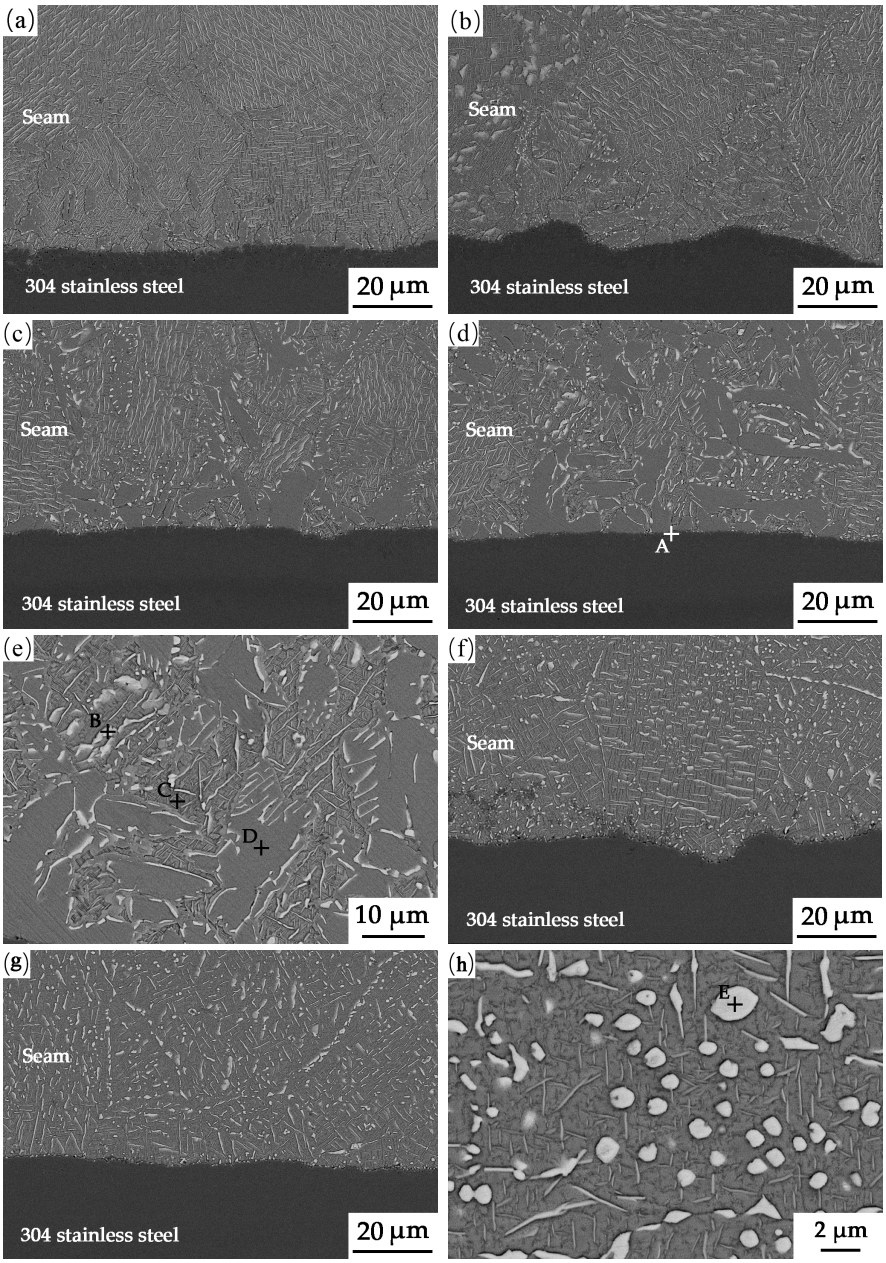

Figure 9. SEM images of microstructures around the interface between 12AgCuZnSn-2In-0.15Pr-*x*Ga filler metals and 304 stainless steel ("*x*": the mass fraction of Ga): (**a**) $x = 0$, (**b**) $x = 0.5$, (**c**) $x = 1$, (**d**) $x = 2$, (**e**) $x = 2$ (high magnification), (**f**) $x = 3$, (**g**) $x = 5$, and (**h**) $x = 5$ (high magnification).

Table 3. EDS analysis of the points indicated in Figure 9 (at.%).

Points	Ag	Cu	Zn	Sn	In	Pr	Ga	Fe	Cr	Ni
A	1.01	2.89	2.49	0.06	0.14	0.02	3.42	70.79	14.87	4.31
B	37.54	31.84	25.06	1.25	3.18	0.06	1.07	-	-	-
C	8.97	51.80	35.36	0.97	1.35	0.07	1.48	-	-	-
D	6.40	58.24	32.21	0.42	0.71	0.04	1.98	-	-	-
E	65.64	9.68	12.91	1.10	10.65	0.06	0.09	-	-	-

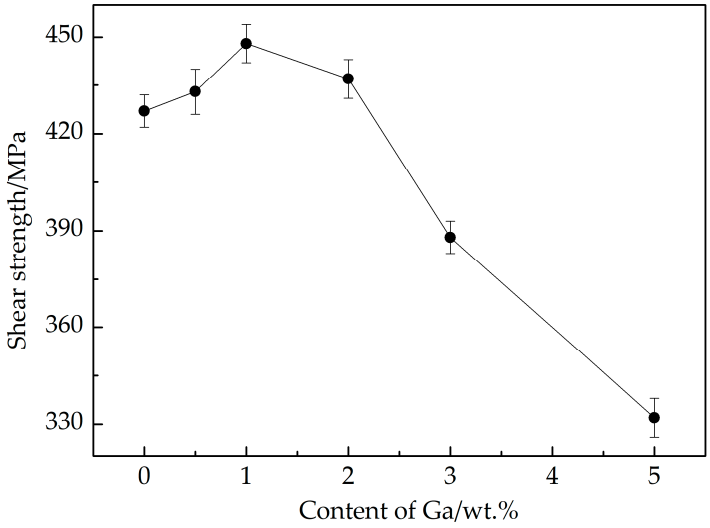

Figure 10. Shear strengths of the 304 stainless steel brazed joints.

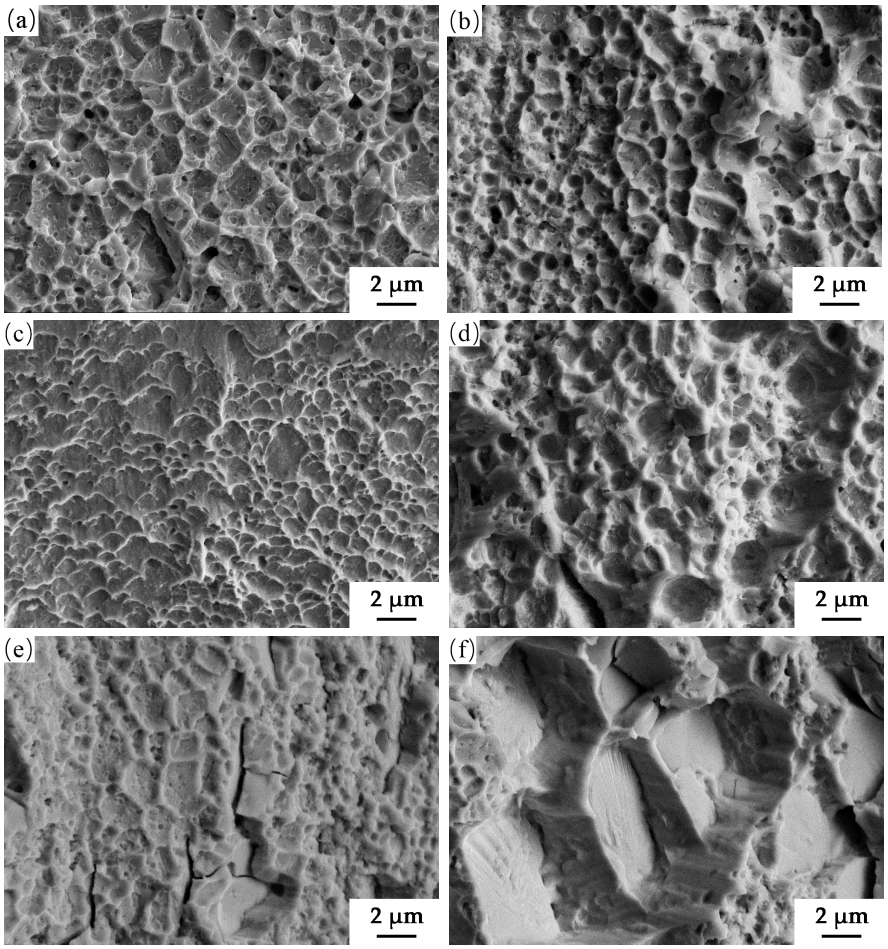

Figure 11. Fracture morphologies of the brazed joints of 12AgCuZnSn-2In-0.15Pr-*x*Ga filler metals ("*x*": the mass fraction of Ga): (**a**) *x* = 0, (**b**) *x* = 0.5, (**c**) *x* = 1, (**d**) *x* = 2, (**e**) *x* = 3, and (**f**) *x* = 5.

4. Conclusions

In this paper, the effect of Ga content on the melting temperature, wettability, and microstructure of 12AgCuZnSn-2In-0.15Pr filler metal was investigated, furthermore, the

mechanical properties and fracture morphologies of the brazed joints were also studied. Based on the experimental results, the conclusions can be drawn as follows:

(1) The solidus and liquidus temperatures decrease with the addition of Ga in the low-silver filler metals, meanwhile, the wettability of 12AgCuZnSn-2In-0.15Pr-*x*Ga is significantly improved. The wetting ring is found on the leading edge of the brazing alloy after the spreading test on 304 stainless steel, which overlaps with the element mappings of Ag, In, and Sn.

(2) The 12AgCuZnSn-2In-0.15Pr-*x*Ga filler metals are mainly comprised of an Ag-based solid solution, Cu-based solid solution, and CuZn compounds. Additionally, the network structure is observed in the filler metal with trace Ga addition. Moreover, some bright Ag-rich phases and gray Cu-rich phases are formed in the matrix when the content of Ga reaches 5 wt.%, and Ga prefers to form a solid solution with Cu, while the In element tends to dissolve into Ag.

(3) Ga has a higher diffusion coefficient into 304 stainless steel than that of Ag, Cu, Zn, etc. The largest shear strength of the brazed joints is obtained when 12AgCuZnSn-2In-0.15Pr-1Ga filler metal was used, and the fracture morphology of the corresponding brazed joint exhibits a much finer and uniform dimple structure than that of the Ga-free one. However, the shear strength of the brazed joints decreases with further increasing Ga content.

Author Contributions: Conceptualization, S.X.; methodology, Q.L.; software, J.W. and L.L.; validation, J.W.; formal analysis, J.W.; investigation, J.W.; resources, J.W.; data curation, J.W.; writing—original draft preparation, J.W.; writing—review and editing, J.W. and P.Z.; visualization, J.W.; supervision, S.X.; project administration, S.X.; funding acquisition, S.X. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest: The authors declare no conflict of interest.

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