SFB 1120

Annual report 2018

Subproject A6

Title

Electron microscopic analysis of melting processes and solidification structures

Project management/-processing

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Task definition

Activities in the current year focus on high-resolution electron microscopic methods for microstructure characterization.
The transformation mechanisms were observed directly in the large chamber SEM (GK-SEM) and investigated in TEM on selected samples.

Procedure

In cooperation with TP A5, soldering ribbons of different composition were characterized with respect to their melting and solidification behavior by heating tests in the GK-REM. The optimum heating temperature and the temperature interval for the soldering process were determined for each individual soldering ribbon system. All samples were examined post-mortem in GK-REM by means of EDX from above and in cross section. The subsequent in-depth TEM investigation provides, among other things, spatially resolved information on the resulting microstructure modifications. For these TEM investigations, lamellae from various selected areas were prepared using a Focused Ion Beam (FIB).

Results

In situ investigations in GK-REM

In 2018 a variation of wrought Al alloys with a variation of Sn solders with different components of other alloying elements was investigated with regard to their wetting behaviour in the heating module of the GK-REM. Table 1 gives an overview of the test program.

Versuch	Basis	Lot	T-Basis	T oberhalb Lot	Kommentar
1.1.1	EN AC-42100	Cu50Sn	420°C 440°C	210°C 225°C	Heizversuch + EDX +EDX Querschliff
1.1.2	EN AC-42100	Cu50Sn	440C°C	320°C	Heizversuch + EDX +EDX Querschliff
1.1.3	EN AC-42100	Cu50Sn	440°C 450°C 460°C	223°C 228°C 231°C	Heizversuch + EDX +EDX Querschliff; TEM
1.1.4	EN AC-42100	Cu50Sn	440°C 460°C 550°C	225°C 233°C 320°C	Heizversuch + EDX +EDX Querschliff
1.1.5	EN AC-42100	Cu50Sn	550°C	310°C	Heizversuch + EDX +EDX Querschliff
2.1.1	EN AW-3003	Cu50Sn	440°C 460°C 480°C 500°C	260°C 254°C 260°C 292°C	Heizversuch + EDX +EDX Querschliff
2.1.2	EN AW-3003	Cu50Sn	440°C 460°C 480°C	225°C 241°C 268°C	Heizversuch + EDX +EDX Querschliff
2.1.3	EN AW-3003	Cu50Sn	550°C	373°C	Heizversuch + EDX +EDX Querschliff
3.1.1	EN AW-4343	Cu50Sn	440°C	270°C	Heizversuch + EDX +EDX Querschliff
3.1.2	EN AW-4343	Cu50Sn	440°C	304°C	Heizversuch + EDX +EDX Querschliff
3.1.3	EN AW-4343	Cu50Sn	440°C	324°C	Heizversuch + EDX +EDX Querschliff
1.2.1	EN AC-42100	Sn	550°C	418°C	Heizversuch + EDX +EDX Querschliff
2.2.1	EN AW-3003	Sn	550°C	360°C	Heizversuch + EDX +EDX Querschliff
3.2.1	EN AW-4343	Sn	550°C	411°C	Heizversuch + EDX +EDX Querschliff
3.2.2	EN AW-4343	Sn	400°C	321°C	Heizversuch + EDX +EDX Querschliff
Probe1	EN AC-42100	Al46Ge10Sn	450°C		EDX (Heizversuch im IOT)
Probe2	EN AC-42100	Al46Ge10Sn	460°C		EDX (Heizversuch im IOT)
Probe3	EN AC-42100	Al46Ge10Sn	500°C		EDX (Heizversuch im IOT)
1.3.1	EN AC-42100	Cu5Sn95	550°C	377°C	Heizversuch + EDX +EDX Querschliff
2.3.1	EN AW-3003	Cu5Sn95	530°C	373°C	Heizversuch + EDX +EDX Querschliff
2.3.2	EN AW-3003	Cu5Sn95	550°C	400°C	Heizversuch + EDX +EDX Querschliff
3.3.1	EN AW-4343	Cu5Sn95	530°C	392°C	Heizversuch + EDX +EDX Querschliff; TEM
1.4.0	EN AC-42100	Cu20Sn75Ge5	390°C		Heizversuch
1.4.1	EN AC-42100	Cu20Sn75Ge5	470°C		Heizversuch
1.4.2	EN AC-42100	Cu20Sn75Ge5	450°C		Heizversuch
1.4.4	EN AC-42100	Cu20Sn75Ge5	450°C	333°C	Heizversuch + EDX +EDX Querschliff

List 1: Overview test program 2018

In the following, experiments 1.1.3 and 3.3.1 will be described in detail, because in addition to the heating experiment and analysis in GK-REM, further investigations in TEM were also carried out.

Experiment 1.1.3

The wrought Al alloy EN AC-42100 was heated to 460°C with the solder Cu50Sn and held at this temperature for about 30 minutes. The solder did not shrink to a spherical shape, but retained its original shape throughout the heating period. Above

440°C the solder began to segregate. At the end of the test it was found that the former solder particle was firmly bonded to the surface.

Figure 1 shows the EDX analysis after completion of the heating test.

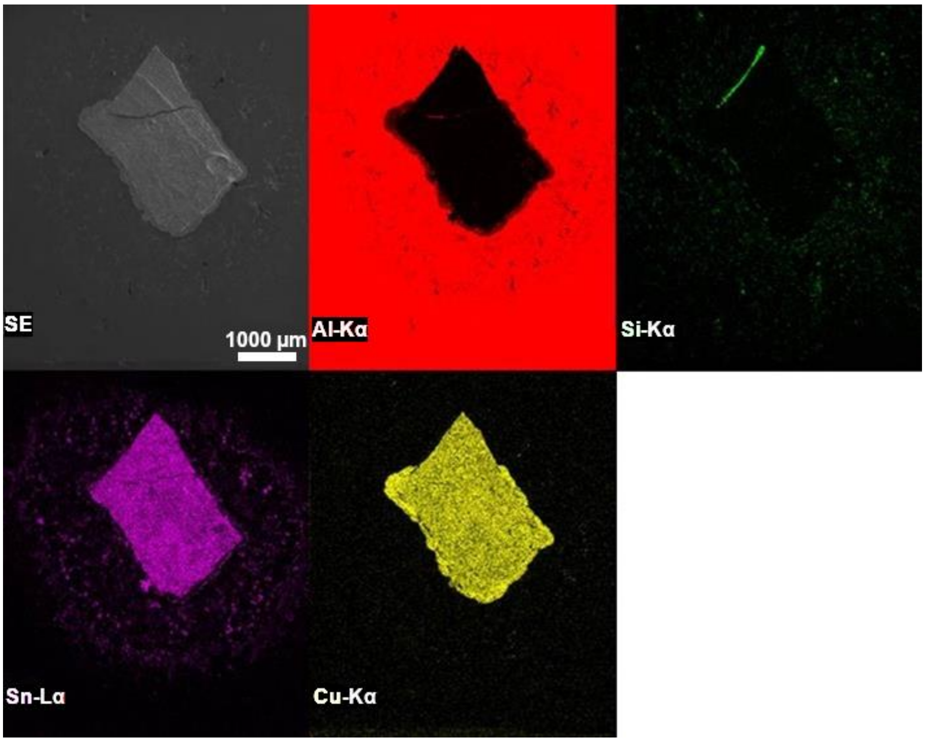

Fig.1: EDX element analysis Test 1.1.3.

Only a small proportion of Cu from the solder alloy has spread onto the base material. This is much more the case for Sn, which can be detected distributed in a circle around the solder.

The sample from test 1.1.3 was ground crosswise in the next step and also examined by means of EDX in the GK SEM (Figure 2).

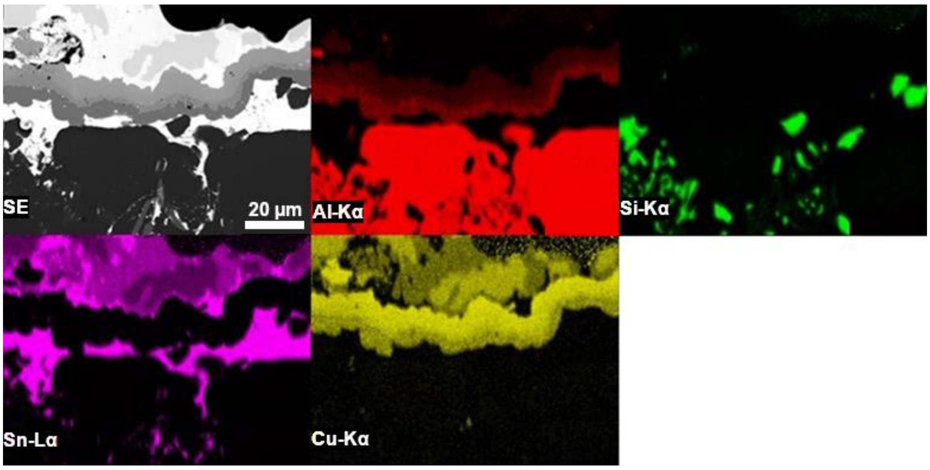

Fig.2: EDX element analysis Test 1.1.3 im Querschliff; 1000x

The cross section clearly shows that Sn has migrated into the base material, while Cu is mostly detected on the surface and has formed a layer in combination with Al. Only small amounts of Cu can be detected in the base material. Sn seems to have migrated into the base material along the Si coated Al grain boundaries.

Experiment 3.3.1

The wrought Al alloy EN AW-4343 was heated to 530°C with the solder Cu5Sn95 and held at this temperature for approx. 30 minutes. The solder shrank to a spherical shape and began to segregate above 500°C. The former shell of the sphere remained intact until the end of the experiment.
Figure 3 shows the EDX analysis performed post mortem in GK-REM.

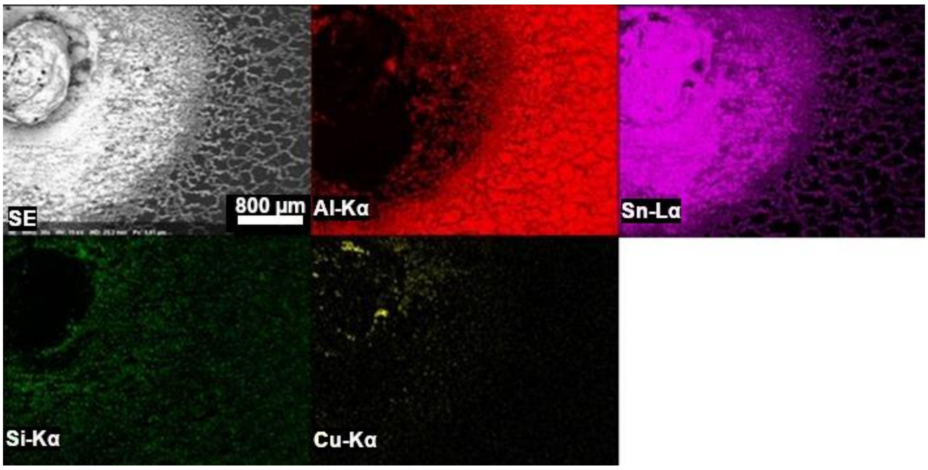

Fig.3: EDX element analysis Test 3.3.1

In the upper left quadrant you can see the shell of the former plumb bob. Around this shell the solder has spread out and melted in a ring around the soldering point. The amount of fluxed material decreases away from the soldering point towards the outside of the picture. During the experiment, the formation of hot cracks and the healing of the cracks could be seen. For Sn one can see in the outer areas of the propagation that it has spread beyond the Si covered Al grain boundaries.

In the next step, the specimen was ground crosswise and examined by means of EDX in GK-SEM (Figure 4).

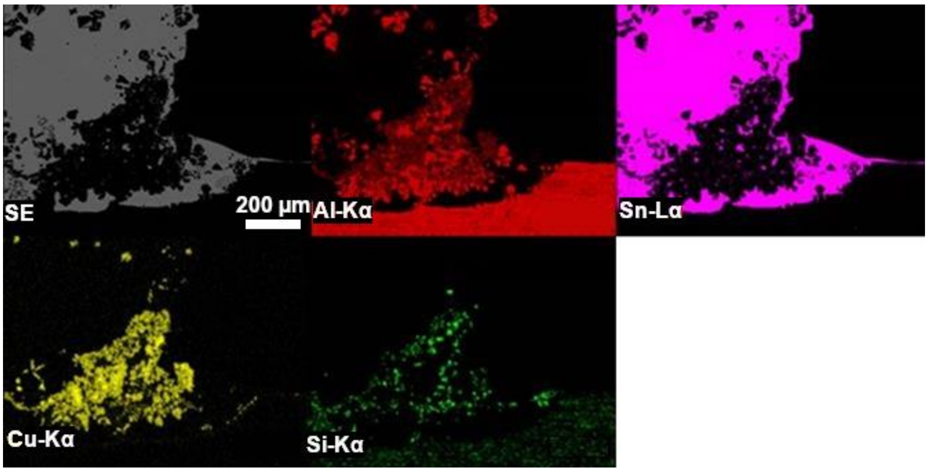

Fig.4: EDX element analysis Test 3.3.1 im Querschliff; 100x

The spherical structure of the former drop of solder is clearly visible. At the junction of solder drops and base material, a mixing of base and solder can be seen. Al and Si have migrated into the solder droplet. The mixing takes place down to a depth of almost 200µm of the base material. Here the solder components Sn and Cu can be detected.

More detailed information on the samples listed here can be obtained from the investigations in the TEM.

TEM Studies

From each sample from experiments 1.1.3 and 3.3.1, 3 lamellae were prepared at different locations using FIB (Figure 5).

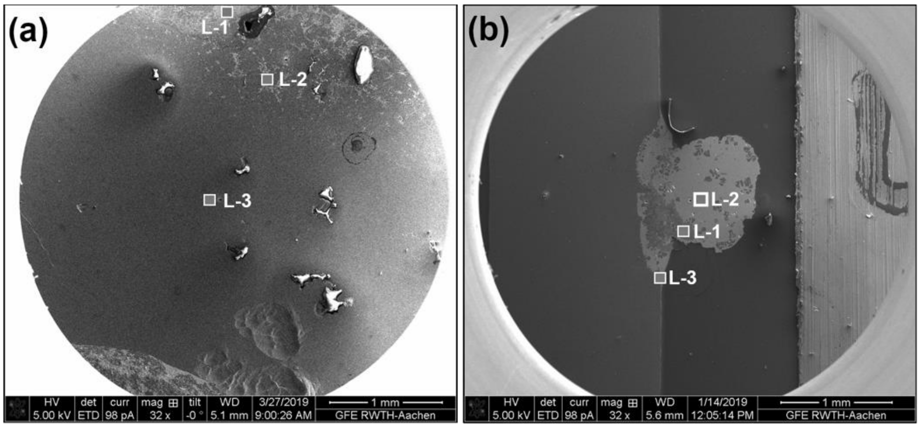

Fig.5: The FIB lamella sampling point of (a) Probe 1.1.3 und (b) 3.3.1.

Figure 6 shows the ADF-STEM image and the corresponding EDX element distribution maps of a FIB section from the center of the solder drop (see L-1 in Figure 5(a)). The EDX elemental distribution maps show an enrichment of the Sn to a depth of about 500 nm. In addition, pores, which probably consist of Al2O3, were detected in this area.

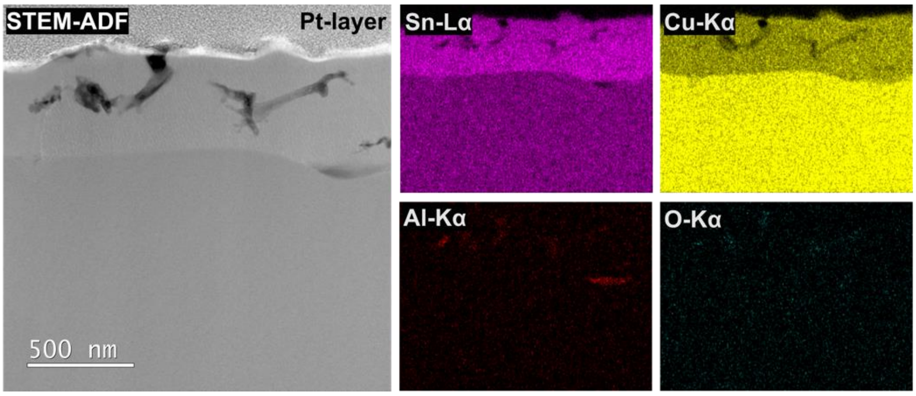

Fig.6: ADF-STEM Figure and associated EDX elemental distribution maps a FIB lamella from the center of the solder drop of sample 1.1.3.

In contrast, no copper can be detected in the upper area in the middle of the solder droplet of sample 3.3.1 (L-2 in Figure 5(b)) (see Figure 7). Pores are also present in the upper part of the sample. In the middle of the solder droplet of sample 3.3.1. no copper can be detected in the upper part (see figure 7). The pores in the upper area, which also have a low content of Al and O, can also be detected here..

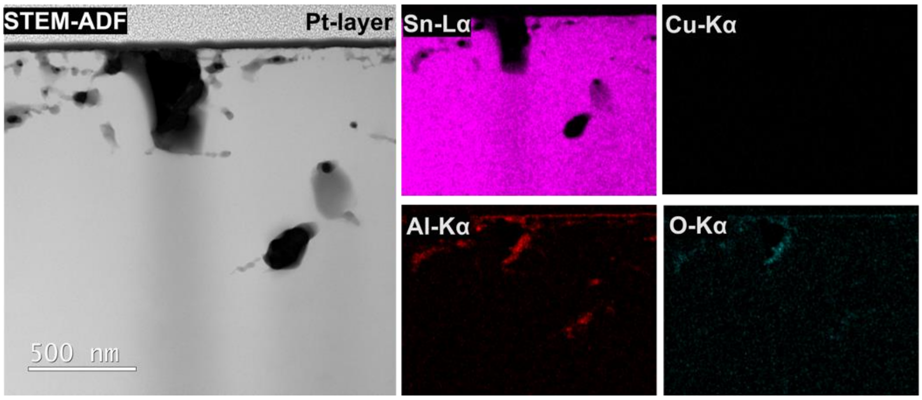

Fig.7: ADF-STEM Figure and corresponding EDX element distribution maps of a FIB lamella from the middle of the solder drop of sample 3.3.1.

Figure 8 shows the ADF-STEM image of the FIB lamella from the outside of the solder drop of sample 1.1.3 (L-2 in Figure 5(a)). The EDX elemental distribution maps for Sn show an enrichment which is only on the surface. Cu cannot be detected in this area. The mapping for Al and O shows for both elements an enrichment only in smaller areas and not along the surface.

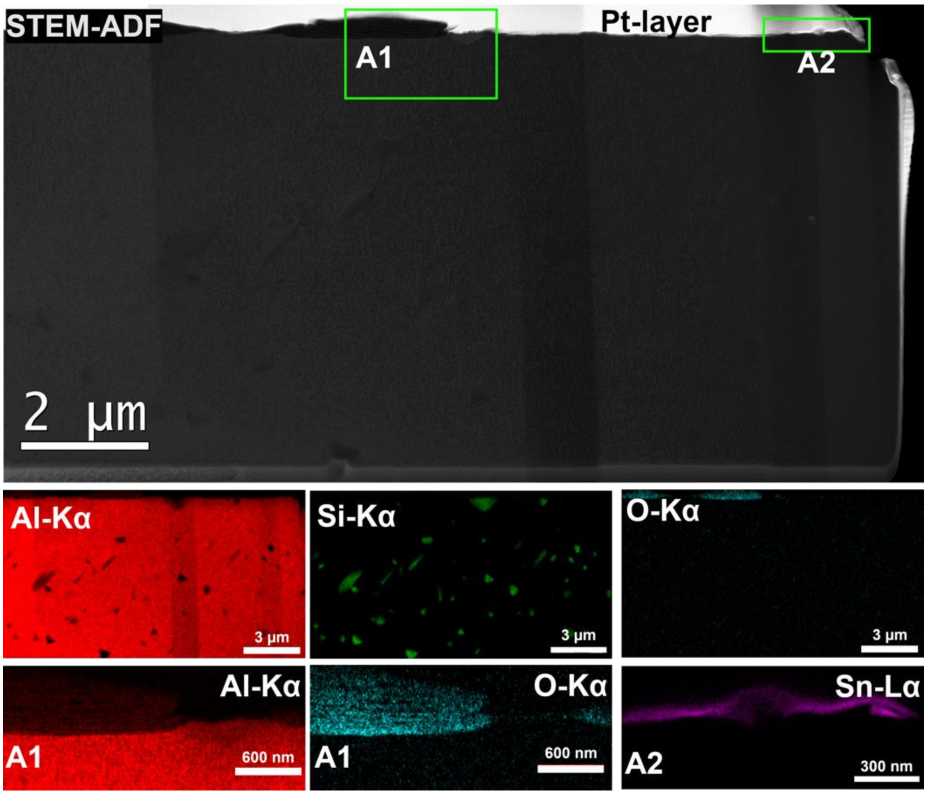

Fig.8: ADF-STEM image and corresponding EDX element distribution maps of a FIB lamella in the outer area of the solder drop of sample 1.1.3.

Summary and Conclusion

As a summary of the results so far:

1. The wetting behaviour of the solders could be successfully observed by in situ heating experiments with the solder system Sn-Cu.
2. The wetting behaviour of the solder can be detected up to the outer area of the solder drop (L-2 in Fig.5.b and Fig.8) The enrichment of the solder component Sn can be detected down to a layer of approx. 100 nm thickness.
3. Wetting was carried out without formation of the native oxide layer. The native oxide layer, in this case Al2O3, could not be detected by ADF-STEM images and EDS analysis. From this we conclude that wetting took place without formation of an oxide skin layer.

Further investigations for the detailed characterization of secondary phases will follow. Furthermore, it is planned to carry out a cooperation with TP B6 for the measurement and simulation of contact angles.

Publications

Aretz, A.; Ehle, L.; Haeusler, A. et al.: In Situ investigation of production processes in a Large Chamber Scanning Electron Microscope, Ultramicroscopy. 193 (2018), S. 151–158. DOI: 10.1016/j.ultramic.2018.07.002