

Application Note # EBSD-04

Identification of complex phases in strontium modified silicon alloys

When analyzing multiphase samples, the QUANTAX EBSD system helps attain excellent quality datasets while optimizing time spent at the scanning electron microscope (SEM). Bruker has developed a semi-automatic Advanced Phase Identification (ID) procedure to overcome the limitations of manual phase identification, known for being time-consuming and often inaccurate.

In this application note, discover how this unique feature combined with ultrafast re-indexing allows characterizing a sample without knowing all present phases during measurement. This is particularly useful if some phases cannot be distinguished by atomic contrast imaging. The dataset can be subsequently completed (and/or corrected) without the need of the SEM. This procedure is performed by finding the best phase entry in the databases, improving both indexing rate and angular resolution.

By combining the chemical and crystallographic information of a phase, the software can automatically identify the best phase fit within the available EBSD databases, i.e. the Inorganic Crystal Structure Database (regularly updated), Crystallography Open Database, American Mineralogist Crystal Structure Database, Bruker database, and any user database.

Advanced Phase ID is available online at the SEM as well as offline. It is not necessary to save diffraction patterns since Kikuchi band positions are automatically saved and generally provide sufficient information.

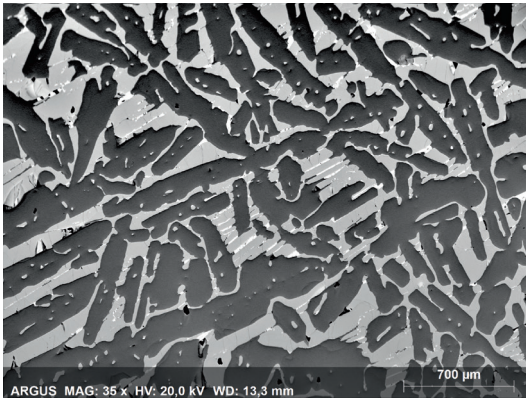
Methodology

First, a combined EDS/EBSD dataset or point measurement is acquired. The software then uses the chemical data obtained by the EDS detector to search all possible candidate phases within the available crystallographic databases. These candidates will be automatically tested to the corresponding experimental pattern (or Kikuchi band position) and the solutions will be classified according to the best fit. This procedure can test several hundred phases in less than 10 seconds and is not limited by a maximum number of candidates. The user can simply add the best fitting phase into the phase list for subsequent ultrafast re-indexing.

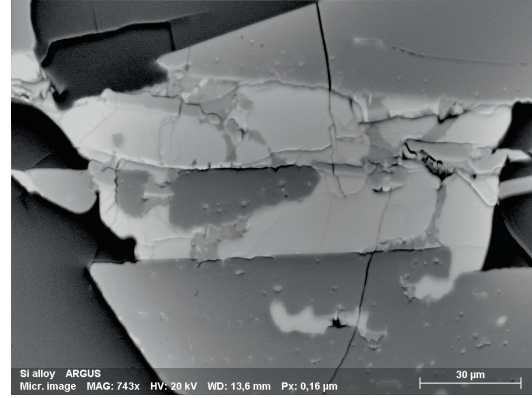
Sample

The investigated sample is a strontium modified aluminum-silicon alloy as shown in Fig. 1. Such casting alloys are

Atomic contrast images



(a)



(b)

Fig. 1 (a) Low magnification atomic contrast ARGUSTM BSE image of the sample; (b) high magnification ARGUSTM BSE image corresponding with the measured area. At least five phases can be distinguished by their atomic contrast.

developed by modifying the eutectic point of aluminum-silicon in order to enhance the material properties. Since the specimen contains some “incidental impurities”, such as iron, it might include phases which are unknown to the user. Furthermore, these phases may not be described in commercial databases.

Measurement conditions

The sample was measured using a Field Emission Gun SEM (FEG SEM) with an XFlash® detector of 30 mm² active area and an e-Flash HR⁺ EBSD detector.

- Acceleration voltage: 20 kV
- Probe current: ~ 4 nA
- Exposure time: 10.9 ms
- Reanalysis speed: 1292 pps
- Total map size: 156.4 x 117.3 μm²
- EBSP resolution: 200 x 150 pixel
- Hit rate: 96.8 %
- Average grain size: 10.87 μm

The sample was mechanically polished with a final stage of 0.04 μm colloidal silica polishing. The ARGUSTM imaging system was used to assess the surface quality and to find a convenient area of interest for analysis. The presence of at least 5 different phases can be inferred from the high magnification atomic contrast ARGUSTM image shown in Fig. 1.

Results

To optimize time spent at the SEM, the unknown phases are subsequently identified using the Advanced Phase ID feature. Thereby, a simultaneous EBSD/EDS measurement was performed using the only known phase silicon. The resulting map is displayed in Fig. 2.

EBSD Phase distribution map after measurement

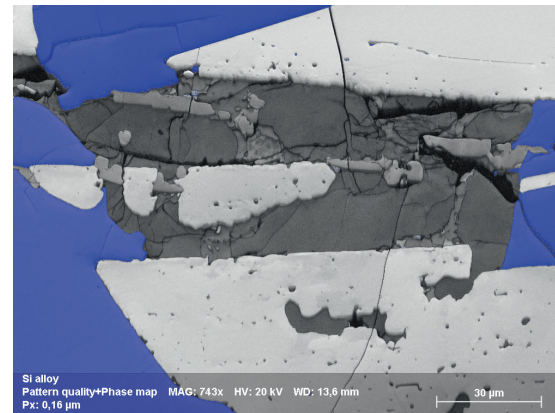


Fig. 2 Pattern quality map (PQM, as background image) with the phase distribution map as seen after measurement. Only the known phase silicon (in blue) was used for calibration and measurement.

Based on the acquired EBSD/EDS dataset, an offline phase identification was performed following the Advanced Phase ID procedure:

1. First, a point was selected from the unindexed areas, identifiable from the phase and PQM maps as seen in Fig. 2.
2. According to its corresponding EDS spectrum shown in Fig. 3a, the ESPRIT software has identified the candidate phases; using the three present elements, i.e. Sr, Al and Si, 11 phase file entries were found out of about 500,000 entries in the commercial databases.
3. Within one second, the ESPRIT software has determined the best solution fitting the corresponding pattern despite its poor quality (Fig. 3b) with 11 fitted Kikuchi bands out of the 12 detected, and a low band mismatch value (“BMM”) of 0.73°, i.e. an excellent fit.
4. The strontium aluminium silicide phase Sr₃Al₁₆Si₃₀ (cubic – IT # 223 – P 42/m 3 2/n) has been added to the phase list.

Phase identification of strontium aluminum silicide

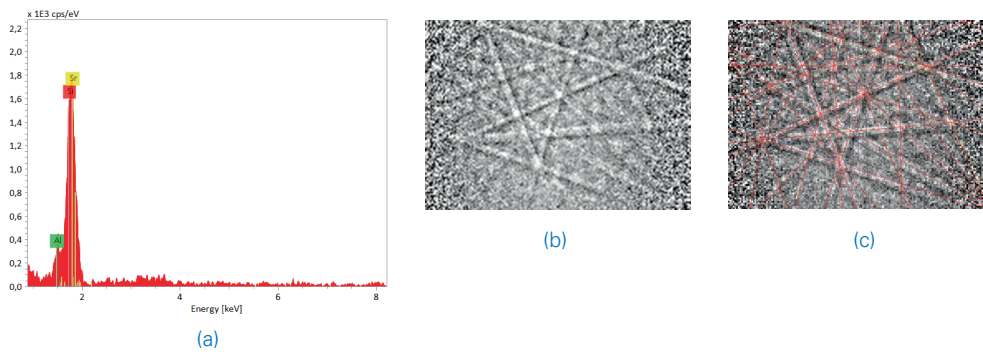


Fig. 3 (a) EDS spectrum of the strontium aluminum silicide phase; (b, c) corresponding diffraction pattern extracted from the measurement (b) and its Advanced Phase ID solution with indexed Kikuchi bands in red overlay (c).

5. The procedure was repeated for all missing phases. The map can then be completed by ultrafast re-analysis using the final phase list. The resulting completed map is displayed in Fig. 4. A total of 8 phases were identified.

Most notable in the investigated sample is the “dysprosium iron silicon” phase (turquoise grains in Fig. 4). The corresponding phase in the sample is actually not defined in the currently available EBSD databases (ICSD, COD, AMCSD, Bruker Database). Nonetheless, Advanced Phase ID has identified a phase file which perfectly fits the crystallographic parameters but not the exact chemistry. However, this approach obtains valuable information, such as orientation and grain size distributions, even though the exact phase file is not available.

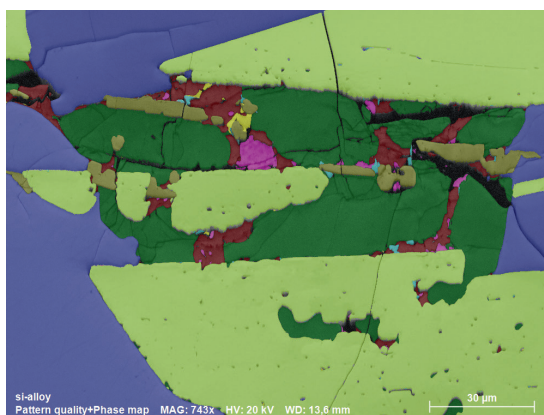
The dataset could easily be reanalyzed in the future if the correct phase file (i.e. with exact chemical and crystallographic information) becomes available.

In order to identify the “dysprosium iron silicon” phase, the candidate phases were first selected using the three present elements Fe, Si and Al identified in the EDS spectrum in Fig. 5a. As a result, 25 phase file entries were found in the databases. However, none of them gave satisfactory fitting results of the corresponding pattern in Fig. 5b.

Consequently, phase search was extended using Fe, Si and any other elements, giving 8407 candidate phases. Within 13 seconds, the Advanced Phase ID procedure had identified dysprosium iron silicide $Dy_{1.17}Fe_4Si_{9.94}$ (hexagonal – IT # 194 – $P6_3/mmc$) as the best phase file fitting the diffraction pattern with 12 out of 12 Kikuchi bands and a low BMM of 0.86° (Fig. 5c).

Despite the lack of dysprosium within the sample, the crystal structure of the dysprosium iron silicide phase file fits the present aluminum iron silicide phase, allowing for analysis of the latter’s microstructure. The excellent quality of the indexing results can be seen in phase and orientation distribution maps (Figs. 4, 6).

Unprocessed EBSD Phase distribution map after re-indexing



No.	Color	Phase name	IT no.	Space group	Crystal system
1	Blue	Silicon	227	Fd3m(*)	cubic
2	Light Green	Ferdisilicite	123	P4/mmm	tetragonal
3	Dark Green	Strontium silicide (1/2)	212	P4 ₃ 32	cubic
4	Purple	Aluminum silicon strontium (1/1/1)	191	P6/mmm	hexagonal
5	Yellow	Strontium silicide (4/7)	141	I4 ₁ /amd(*)	tetragonal
6	Turquoise	Dysprosium iron silicon (1.2/4/9.8)	194	P6 ₃ /mmc	hexagonal
7	Olive Green	Titanium(II) iron silicide (1/1/2)	55	Pbam	orthorhombic
8	Dark Red	Strontium aluminum silicide (8/16/30)	223	Pm3n	cubic

Fig. 4 Completed EBSD Phase map after re-indexing with the PQM as background. The phase distribution is given in the table. Reanalysis time was less than 10 minutes, i.e. 1300 pps using a laptop. Non-indexed points belong to the porosity and are visible in black.

Phase identification of iron aluminium silicide

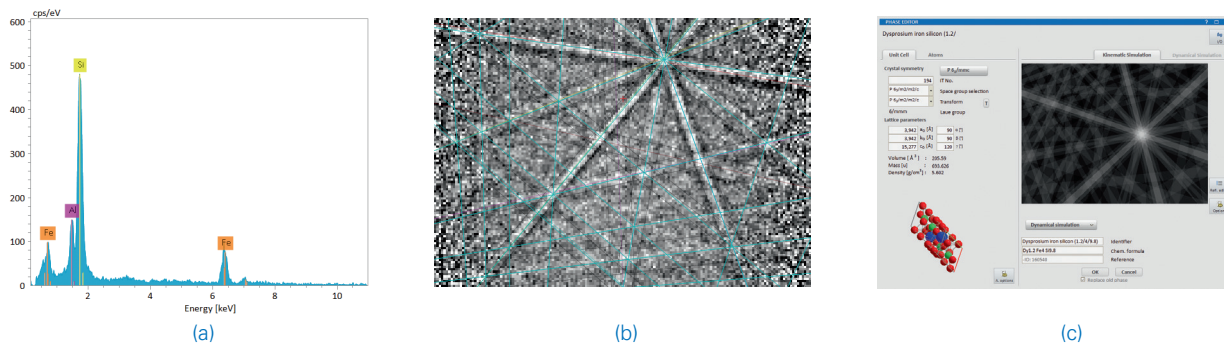


Fig. 5 (a) EDS spectrum of the iron aluminium silicide phase; (b) corresponding diffraction pattern (extracted from the measurement) with the solution as dysprosium iron silicide phase with the indexed bands displayed in blue overlay; (c) corresponding crystallographic information with unit cell and spherical Kikuchi pattern (kinematic simulation).

Completed Orientation distribution map

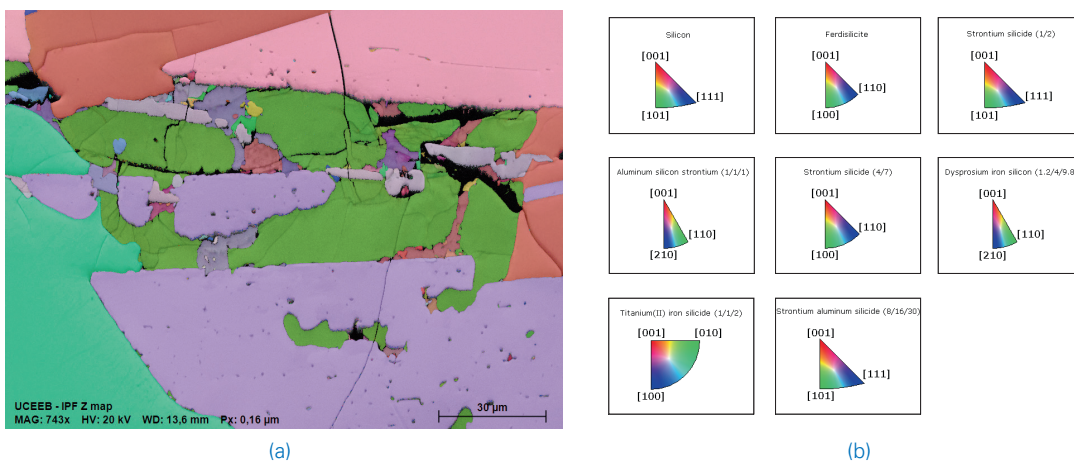


Fig. 6 (a) Unprocessed orientation distribution as Inverse Pole Figure map along the Z axis (perpendicular to the sample surface) with PQM as background; (b) corresponding centrosymmetric Laue group specific coloring scheme for each crystallographic phase.

Conclusion

The Advanced Phase ID feature available in the ESPRIT software is a highly performant, semi-automatic phase identification procedure which efficiently combines the information simultaneously acquired by EBSD and EDS techniques. It allows to considerably optimize time spent at the SEM by conducting simultaneous EBSD/EDS measurements with the known phase(s) only and by performing subsequent offline phase identification before ultrafast re-indexing to complete or correct the analysis.

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