The AlFe2Cr6Si1Ti1.5 alloy of intended composition was prepared by melting of pure aluminium and silicon with Al–Cr, Al–Ti, and Al–Fe master alloys in an electric resistance furnace in argon atmosphere. In the next step, the AlFe2Cr6Si1Ti1.5 alloy was melted in an induction furnace and gas-atomized by high-pressure nitrogen.
(500 kPa), forming rapidly solidified powder. Prepared powder was sorted into fractions with particle sizes: under 63 µm, 63-125 µm and above 125 µm. It can be expected that each fraction has been solidified with different quenching rate, which is reflected in phase composition of each fraction mainly on content of metastable phases. There is also different surface to volume ratio of the particles, which has an influence of the sintering conditions. Samples of each powder fraction were compacted by spark plasma sintering method at temperature of 500°C for 15 min. The pressure was set at 48 MPa and the sintering was carried out in continuous mode.

Microstructure of compact samples (after grinding, polishing and etching by hydrofluoric acid with concentration of 0.5 vol.%) was observed by the metallographic optical microscope Olympus PME3. Residual porosity of sintered materials was measured using Image J software from optical micrographs. Microstructure of powder samples was observed by SEM TESCAN VEGA 3 LMU with BSE detector. The phase composition was evaluated using a PANalytical X’Pert Pro X-ray diffractometer using the HighScore Plus software and PDF2 database. Thin foils for TEM were prepared by grinding until final thickness down to 50–80 µm and by subsequent electrolytic polishing in mixture of HNO₃ and methanol (1:3) at –20 °C with final ion polishing by Gatan PIPs. The samples were investigated by transmission electron microscope EFTEM JEOL 2200 FS. Compression tests on compact samples were performed using testing machine LabTest 5.250SP1-VM. Cubic samples with size of 3 mm were used. The testing direction of compression tests was similar to uniaxial pressing direction during compaction of bulks. Each compression experiment was repeated 3 times.

3. Results and discussion

Microstructure of different powder fractions of AlFe2Cr6Si1Ti1.5 alloy is given in Fig. 1. The smaller the particles are, the finer the microstructure is. It is in agreement with assumption that smaller particles achieve higher cooling rates during solidification process.

Microstructure of compact AlFe2Cr6Si1Ti1.5 alloys from various powder fractions are shown in Fig. 2. XRD patterns of AlFe2Cr6Si1Ti1.5 alloys from various powder fractions are shown in Fig. 3. All powder fractions contained α-Al, stable intermetallic phase Al₃Cr₂ and quasicrystalline phase Al₈₄Cr₁₅ described as icosahedral in [11]. The Al₈₄Cr₁₅ quasicrystalline phase described in [12] was observed only in rapidly solidified powder with the finest particle size fraction (under 63 µm) due to highest quenching rate of this fraction enabling to form highly alloyed metastable phase. Residues of this phase were detected in the compacted sample prepared from the finest powder, but the majority was decomposed during compaction by SPS. Evolution of Al₃Fe₄ phase during compaction can be also observed in Fig. 3. This phase is formed by decomposition of both super-saturated solid solution of Fe in Al matrix and Al₈₄Cr₁₅ quasicrystalline phase. At elevated temperatures quasicrystalline phases are transformed into more thermodynamically stable crystalline intermetallic phases (mainly Al₃Cr₂ and Al₃Fe₄) [4]. This transformation is accompanied by increase of size of intermetallic particle and by change of particles morphology [3]. We assume that this process can lead to closing of crack in the material and so provide the material with “self-healing” properties.

![Fig. 1. Microstructure of AlFe2Cr6Si1Ti1.5 powder alloy with fractions: (a) under 63 µm, (b) 63–125 µm, and (c) above 125 µm (SEM).](image-url)
Fig. 2. Microstructure of AlFe2Cr6Si1Ti1.5 alloy sintered from powder fraction: (a) under 63 µm, (b) 63–125 µm, and (c) above 125 µm (LOM).

Fig. 3. Detailed XRD pattern of AlFe2Cr6Si1Ti1.5 alloy (phases: 1 — Al, 2 — Al95Fe4Cr1, 3 — Al13Cr2, 4 — Al84Cr15, 5 — Al13Fe4).
No Ti- and Si-containing phases were identified in the XRD patterns, probably due to a small amount of these elements in the initial powders or close atomic radii of alloys elements and so dissolving of the titanium in quasicrystalline phases or aluminium (dissolving of the silicon in phase with iron). This assumption is in agreement with data reported by Kimura et al. [4], who described various ternary Al–Fe–Cr–Ti quasicrystalline phases.

The microstructures of compacted AlFe2Cr6Si1Ti1.5 alloys were investigated and documented by transmission electron microscope (Fig. 4). The microstructures of alloys composed of finely dispersed particles of intermetallic phases (IP — darker parts) in the α-Al matrix (lighter parts). The intermetallic particles are located predominantly at the grain boundaries, frequently they are presented in form of clusters. It complicates their better description e.g. by SAED by overlaying individual particles and matrix. The main difference among materials prepared from various initial particle size is the grain size of the aluminium matrix, which is given in Table I.

The average size of coherently diffraction domains of aluminium (Table I) estimated from XRD patterns by the Scherrer calculator is about 0.1 μm for all materials. The average grain size measured from TEM images is between 1 and 2.6 μm. This disagreement of obtained values can be explained by the fact that from XRD the size of coherently diffracting domains is estimated, which corresponds e.g. to subgrain size. On the other hand, the value determined from TEM images corresponds to the grains size. In our case, the subgrains are not visible by TEM, and the difference can be caused by presence of clusters of intermetalics on the grain boundaries distorting the matrix lattice or by measuring error. Due to submicrometer scale, it is not possible to prove the presence of subgrains by EBSD.

Mechanical properties of material prepared by powder metallurgy are influence by two contradictory factors. The first one is grains size. With decrease of grain size, the values of yield stress should be higher according to the Hall–Petch relationship. The other factor is presence of oxide layers on the surface of initial powder particles. They could serve as strengthening particles (so-called oxides dispersion strengthening = ODS). More likely, they form the week parts at grain boundaries and so decrease the cohesion of sintered particles. Therefore, the effect of oxide particles is not unambiguous. The values of compressive yield strength of compacted alloys are shown in Table II. The values are similar within ex-

<table>
<thead>
<tr>
<th>Particle size fractions (μm)</th>
<th>Powder Al subgrain size (XRD) [nm]</th>
<th>Bulk Al subgrain size (XRD) [nm]</th>
<th>Bulk Al grain size (TEM) [μm]</th>
<th>Yield strength in compression [MPa]</th>
</tr>
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<tbody>
<tr>
<td>under 63</td>
<td>60 ± 20</td>
<td>100 ± 30</td>
<td>1.0 ± 0.2</td>
<td>242 ± 4</td>
</tr>
<tr>
<td>63–125</td>
<td>100 ± 20</td>
<td>130 ± 30</td>
<td>1.9 ± 0.3</td>
<td>246 ± 4</td>
</tr>
<tr>
<td>above 125</td>
<td>100 ± 20</td>
<td>120 ± 30</td>
<td>2.6 ± 1.3</td>
<td>235 ± 12</td>
</tr>
</tbody>
</table>

Fig. 4. TEM cross-section micrographs (Al and intermetallic phases Al13Cr2, IP) are marked of the RS Al–Fe–Cr alloys compacted from particle size fractions: (a) under 63 μm, (b) 63–125 μm and (c) above 125 μm.
perimental errors. It means that no strengthening due to the Hall–Petch relationship was observed for the material prepared from the finest powder fraction. It can be explained by presence of highest amount of oxide particles. They do not cause ODS but are probably the weak parts of sintered material. Nevertheless, materials prepared in our research had lower yield strength as analogous materials published in literature [5, 6] but higher than material prepared from the same powder by hot extrusion [3].

<table>
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<tr>
<th>Alloy composition</th>
<th>Yield strength [MPa]</th>
<th>Reference</th>
</tr>
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<tbody>
<tr>
<td>AlFe2Cr6Si1Ti1.5</td>
<td>120</td>
<td>[3]</td>
</tr>
<tr>
<td>Al95Cr3.1Fe1.1Ti0.8</td>
<td>547</td>
<td>[5]</td>
</tr>
<tr>
<td>Al93Fe3Cr2Ti2</td>
<td>544</td>
<td>[6]</td>
</tr>
<tr>
<td>Al92.5Ti2.5Fe2.5Cr2.5</td>
<td>540</td>
<td>[6]</td>
</tr>
</tbody>
</table>

Materials prepared from different powder fraction have also different residual porosity that was 0.6 vol.% for the finest fraction, 2.8 vol.% for the fraction 63–125 μm and 4.0 vol.% for the fraction above 125 μm. The increase of porosity can be explained by presence of hard oxide layers on the surface of initial particles that do not allow particles to deform during sintering. Because of that the filling is better with smaller particles.

4. Conclusion

The AlFe2Cr6Si1Ti1.5 alloy was successfully prepared (in form of bulk materials, with low residual porosity) by powder metallurgy using melt atomization and spark plasma sintering under the following conditions: temperature of 500°C and sintering time for 15 min. Three different powder granulometric fractions were used for compaction (less than 63 μm, 63–125 μm and above 125 μm). All three alloys prepared in this way were formed by α-Al solid solution with intermetallic phases Al13Cr2 and Al13Fe4 and quasicrystalline intermetallic phase Al95Fe13Cr1. Moreover, the finest fractions of powder contained quasicrystalline intermetallic phase Al95Cr15, that it was partially decomposed during the compaction process forming stable intermetallic phases. By room temperature testing, all alloys exhibited good yield strength comparable with analogous materials. The best value of yield strength was obtained for the material prepared from powder fraction 63–125 μm, but the difference was not significant. The future research will be focused on the alloy prepared from the finest powder fraction as it contains two quasicrystalline phases, which gives the material potential for possible self-healing behaviour.

Acknowledgments

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References