APPLICATION OF X-RAY DIFFRACTION TO ANALYSE PHASE COMPOSITION OF ALUMINIUM ALLOYS FOR PLASTIC WORKING

An attempt was made to determine phase composition of commercial aluminium alloys using X-ray diffraction. Samples for phase composition analysis were selected from the group of aluminium alloys covered by the EN 573-3:2013 standard [1]. Representative samples were taken from eight groups of alloys with different chemical composition (at least one sample from each group). The diffraction intensity was measured with a standard X-ray diffractometer in Bragg-Brentano geometry in a way that allowed identification of the weakest diffraction peaks.

As a result of the performed research it has been shown that X-ray phase analysis can be used to identify the matrix of aluminium alloys, Si and crystalline intermetallic phases such as Mg2Si, Al4.01MnSi0.74, Al93.38Cu6.02Fe24Si16.27, MgZn2, Al17(Fe3.2Mn0.8)Si2, Al65Cu20Fe15, and Cu3Mn2Al. The detectability limit of the above-mentioned phases is better than 0.5%. The research has also shown that X-ray phase analysis is applicable in the investigation of phase transformations taking place in aluminium alloys.

Keywords: Aluminium alloys, X-ray diffraction, qualitative phase analysis

1. Introduction

Aluminium alloys are widely used in the global economy, mainly due to the favourable relationship between functional properties and production costs. The main alloying constituents used in the production of aluminium alloys are copper, manganese, magnesium, silicon, zinc and iron. All aluminium alloys for plastic processing are suitable for deformation strengthening. Additionally, alloys from the 3000 and 5000 series can be solution hardened (by Mn and Mg respectively dissolved in solid aluminium). In turn, alloys from the 2000, 6000 and 7000 series are dispersion hardened. Various diffraction methods are now considered the best tool for phase identification. Studies of the phase composition of aluminium alloys are based, among others, on the method of X-ray phase analysis, which consists in identification of the features of a crystal structure related to the distribution of diffraction patterns intensity. The X-ray phase analysis uses the phenomenon of X-ray diffraction and a comparison of the empiric diffractogram with a database of powder diffraction patterns.

2. Test materials and methods

To analyse the phase composition, samples of commercial aluminium alloys classified in the PN-EN 573-3 standard [1] were taken. Alloys from the 1000 series described in the standard are sometimes called metallurgical or pure aluminium with an aluminium content exceeding 99%. The 2000 series alloys, also known under the trade name Duralumin, contain several percent of copper, while the 3000 series alloys contain a few percent of manganese. The 4000 series symbols are used in the standard to designate aluminium-silicon alloys with the chemical composition similar to silumins used in foundries. The 5000 series includes aluminium and magnesium alloys, while the 6000 series is composed of aluminium alloys with magnesium and silicon. The 7000 series aluminium alloys are alloys containing several percent of zinc, magnesium and copper. The 8000 series aluminium alloys contain iron and silicon. Phase analysis was conducted on the following aluminium alloys: EN AW1050A, EN AW2017A, EN AW3104, EN AW4045, EN AW5754, EN AW6082, EN AW7075 and EN AW7003, and also EN AW8011. All alloys were in the near-phase equilibrium state. The chemical composition of selected alloys is summarized in Table 1.

The X-ray phase analysis was performed using a standard X-ray diffractometer with an energy-dispersive detector in Bragg-Brentano geometry [2,3]. A sufficiently long measurement time and an optimized aperture set were provided to obtain diffractograms with a satisfactory signal-to-noise ratio. Based on previous own studies, it was assumed that X-ray diffraction would occur in the crystalline fraction of the sample with crystallites larger than a fraction of a micrometre.

As a result of the X-ray diffraction measurements, sets of numbers were obtained, which were next processed into a graphi-
The range of diffractions intensities was selected in such a way that it was possible to identify the reflections of the lowest intensity. The X-ray phase analysis was based on the compatibility of empirical diffractograms with the diffractograms of crystalline substances described in the identification catalogue [4]. To confirm the occurrence of phases identified by X-Ray diffraction conducted chemical composition analysis in micro-areas using the SEM Inspect F50 microscope with an EDS X-ray microanalysis device. For each of the samples, images of microstructure were made in 4000× magnification, in a working voltage of 20 kV. EDS spectra recorded in graphic form and the average percentage of each element were generated using a dedicated Team program.

3. Results and discussion

| EN AW-1050A | 0.25 | 0.40 | 0.05 | 0.05 | 0.05 | 0.07 |
| EN AW-2017A | 0.2-0.8 | 0.7 | 3.5-4.5 | 0.4-1.0 | 0.4-1.0 | 0.25 |
| EN AW-3104 | 6 | 0.8 | 0.05-0.25 | 0.8-1.4 | 0.8-1.3 | 0.25 |
| EN AW-4045 | 9.0-11.0 | 0.8 | 0.3 | 0.05 | 0.05 | 0.1 |
| EN AW-5754 | 0.4 | 0.4 | 0.1 | 0.5 | 2.6-3.6 | 0.2 |
| EN AW-6082 | 0.7-1.3 | 0.5 | 0.1 | 0.1-0.45 | 0.6-1.0 | 0.2 |
| EN AW-7003 | 0.3 | 0.35 | 0.2 | 0.3 | 0.5-1.0 | 5.0-6.5 |
| EN AW-7075 | 0.4 | 0.5 | 1.2-2.0 | 0.3 | 2.1-2.9 | 5.1-6.1 |
| EN AW-8011 | 0.3-1.1 | 0.4-1 | 0.1 | 0.1 | 0.05 | 0.1 |

The X-ray diffraction produced strong reflections from the alloy matrix and weak reflections from other alloying constituents.

As a result of the analysis of the diffractogram of the EN AW1050A aluminium, diffraction reflections that might originate from silicon and an intermetallic phase with the stoichiometry were identified. The EN AW1050A aluminium does not contain deliberately introduced...
alloying additions. Elements such as silicon, iron, copper, manganese, magnesium, zinc and titanium are present in 1050A aluminium as impurities originating from electrolysis or recycling [5]. This means that the impurities present in the EN AW1050A aluminium formed the Al$_{93.38}$Cu$_{6.02}$Fe$_{24}$Si$_{16.27}$ intermetallic compound and free silicon in the crystalline form. The proportions of concentration of elements in Al$_{93.38}$Cu$_{6.02}$Fe$_{24}$Si$_{16.27}$ are roughly in line with the proportions of these constituents in the standard chemical composition of 1050A aluminium.

As a result of the analysis of the diffractogram of the EN AW-2017A aluminium alloy, the crystal structure of the examined alloy was found to be consistent with the crystal structure of the two phases with the Cu$_3$Mn$_2$Al and Mg$_2$Si stoichiometry. From the EN 573-3 standard [1] it follows that the EN AW-
From the 5000 series, alloys designated with the symbol EN AW-7003 and EN AW-7075 were selected for studies of the phase composition. In the 7000 series, alloys designated with the symbols EN AW-6082 and EN AW-6061 were selected for studies of the phase composition. In the 6000 series, alloys designated with the symbol EN AW-6060 were selected for studies of the phase composition. In the 5000 series, alloys designated with the symbols EN AW-5754 and EN AW-5657 were selected for studies of the phase composition. In the 4000 series, alloys designated with the symbol EN AW-4045 were selected for studies of the phase composition. In the 3000 series, alloys designated with the symbol EN AW-3104 were selected for studies of the phase composition. In the 2000 series, alloys designated with the symbol EN AW-2017A were selected for studies of the phase composition. From the 1000 series, alloys designated with the symbol EN AW-1060 were selected for studies of the phase composition. From the 0000 series, alloys designated with the symbol EN AW-0000 were selected for studies of the phase composition.
Al-Zn-Mg-Cu alloy obtained by Rapid Solidification. Using X-ray diffraction methods, the presence of a solid solution of aluminium and MgZn2 was detected [15]. In the alloy designated as EN AW-7075, intermetallic phases with the Al4.01MnSi0.74 and Mg2Si stoichiometry were identified. It was calculated that in the EN AW-7075 alloy in the state of phase equilibrium may contain about 1.1 wt% of Mg2Si. The detectability threshold of the Mg2Si phase in this alloy was estimated at about 0.2%. The possibility of identification of the Mg2Si phase in the 7000 series alloys is known from the literature [18].

From the 8000 series, the EN AW-8011A alloy was selected for studies of the phase composition in two different states, i.e. before and after heat treatment. The heat treatment of the EN AW-8011A alloy was carried out at 600°C for 100 hours and its purpose was to bring the alloy closer to the state of phase equilibrium. On the diffractogram made before heat treatment, reflections from the Al1.17Fe2.3Mn0.8Si2 intermetallic phase and free silicon were identified. This means that in the EN AW-8011A alloy, silicon crystallized in free form (chemically unbound). After heat treatment, the diffraction reflections from the free silicon were present no longer, but the presence of a phase with the Al1.17Fe2.3Mn0.8Si2 stoichiometry was identified. This means that either the silicon dissolved in the intermetallic compound during heat treatment or together with aluminium formed a eutectic which, being a very fine mixture of constituents, has resulted in a significant widening of reflections and disappearance of diffraction. Atoms of iron and manganese have ions of similar dimensions, a similar number of electrons and similar basic physicochemical properties. In intermetallic compounds it is possible to substitute iron atoms with manganese atoms. This phenomenon no major changes in the crystal structure of these phases [19].

4. Summary and conclusions

1. In the examined aluminium alloys, Al, Si and the following intermetallic phases were identified: Al93.38Cu6.02Fe24Si16.27, Al4.01MnSi0.74, Si, Mg2Si, MgZn2 and Al1.17(Fe2.3Mn0.8)Si2 as well as Cu3Mn2AI.

2. The aluminium alloys described in the EN 573-3 standard may contain numerous alloying elements and impurities, which means that in these alloys the state of phase equilibrium is not always compatible with the well-known binary phase equilibrium diagrams.

3. X-ray phase analysis is applicable as a tool to control phase transformations in commercial aluminium alloys.

4. As a result of the conducted studies it was shown that the detectability threshold of X-ray phase analysis depends on the type of substance examined and assumes the value of a fraction of percent. For example, in the EN AW-6082 alloy it is possible to identify the Mg2Si phase at a concentration of about 0.3%.

5. The EDS result is consistent with the chemical composition of the phases identified by X-ray diffraction.

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