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ORIGINAL ARTICLE

Rapidly solidified Al-Cu-Co quasicrystalline alloy: microstructure and catalytic properties

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Abstract
This study presents the characterisation and evaluation of the catalytic potential of an alloy with a composition referring to the quasicrystal from the Al-Cu-Co system. The material was synthesised through a melt-spinning process, providing rapid solidification conditions and a favourable material form of thin ribbons. Microstructural and chemical analyses were performed using scanning electron microscopy, X-ray diffraction and transmission electron microscopy methods. The obtained results confirmed the presence of a major phase which was a decagonal quasicrystal in the form of globular grains and additional crystalline phases occurring along the grain boundaries. The catalytic potential of the materials was explored using phenylacetylene hydrogenation. The catalyst, obtained by pulverising the ribbons, demonstrated catalytic activity with over 40% phenylacetylene conversion and selectivity exceeding 60% for styrene production under mild reaction conditions. The recovered catalyst displayed a stable phase composition, as confirmed by X-ray diffraction, and unchanged morphology, indicating the possibility of catalyst reuse for subsequent reaction cycles. The presented results provide insight into the experimental verification of the catalytic properties of the developed environmentally friendly catalysts.

Keywords Melt spinning · Quasicrystal · Catalysis · Microstructure characterisation · TEM

1 Introduction

Quasicrystals are considered as functional materials for a wide spectrum of applications due to specific properties resulting from nonperiodic order, as well as geometric and electronic structure of clusters [1]. One of the intensively explored areas is the catalytic application of these materials [2]. The main course of investigation concerns quasicrystals as precursors for catalysts with large specific surface areas obtained in the chemical leaching process using acid or alkaline solutions [3]. Till now, Al-Cu-Co [4] and Al-Cu-Fe [5] quasicrystals have been utilised as methanol steam reforming catalysts with satisfactory results [6]. Transition metals and their oxides, like Cu_2O, provide active centres of catalyst. The favourable surface arrangement for catalytic application is preserved from the quasicrystal structure [7].

The other approach uses bulk metallic materials as catalysts [8]. Quasicrystals provide a specific electronic structure that is favourable for catalytic performance in hydrogenation reactions carried out in liquid or gas environments. This topic is explored mainly for approximant phases containing the same atom clusters as decagonal quasicrystals from the Al–Ni–Co system [9]. The catalytic activity was confirmed for $Al_{13}Co_4$ and Al_5Co_2 phases, which could provide almost full substrate conversion and high selectivity in butadiene or phenylacetylene hydrogenation. The catalytic performance of these compounds was linked with the structure of (2–10) Al_5Co_2 [10] and (100) $Al_{13}Co_4$ surfaces [11], which contain pentagonal clusters arrangement providing proper adhesion and reaction conditions. Reactions in the liquid environment were also carried out, but they required a modified approach to provide sufficient specific surface area for the catalytic processes. For this purpose, the catalyst in the form of powder for pulverised melt-spun ribbons was used. The high substrate conversion and stability of the catalyst were proved for approximants from Al-Co and Al–Fe systems [12] and Al-Co quasicrystalline phase [13]. Although investigated

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approximants are structurally related to the decagonal quasicrystal from the Al-Cu-Co system, the catalytic activity of the quasicrystalline phase has not been yet demonstrated.

Al-Cu-Co quasicrystal was discovered in both equilibrium and nonequilibrium conditions in cast and annealed samples or rapidly solidified alloys [14]. The reported range of chemical composition for Al-Cu-Co quasicrystal is quite broad, and single-phase quasicrystalline alloy could be expected in alloys containing Al, 15–20 at.% of Cu and 15–20 at.% of Co [14]. Nevertheless, the Al-Cu-Co quasicrystal forms mainly in the vicinity of approximate phases [15], like cubic Al(Cu, Co) [16] and monoclinic $Al_{13}Co_4$ [17]. The Al-Cu-Co quasicrystal has a decagonal structure isotypic to Al–Ni–Co quasicrystals [18] with periodicity in one direction [19]. The comparison of the bonding character and bond energy for local clusters around the transition metal sites shows the chemical disorder of the Al–Ni–Co quasicrystal and the differences in electronic structures that may impact the varied functional properties of these phases [20]. Another aspect that determines functional properties is their surface composition, which significantly differs from the bulk material structure [21].

Literature reports indicate the applicability of aluminium-based approximants and suggest quasicrystals as effective catalysts in hydrogenation reactions [11]; however, this field has not been widely examined. Ternary quasicrystals containing aluminium and transition metals as a group of stable phases [22] may be a promising option for efficient and cheap catalysts. This work aims to verify the possibility of application as a catalyst ternary alloy with chemical composition and expected phase composition related to phases with high catalytic performance in hydrogenation reactions. For this purpose, an alloy with composition referring to the quasicrystalline phase from the Al-Cu-Co system [23] was manufactured in the melt-spinning process and then characterised in terms of its phase, chemical composition and morphology. Next, the ribbons were pulverised and applied as catalysts for the liquid-environment phenylacetylene hydrogenation reaction under mild conditions. The effect of the catalyst performance was studied by gas chromatography to evaluate the change in the composition of the reaction mixture during the test. After the catalytic experiment, the stability of the catalyst was verified using X-ray diffraction and electron microscopy observations.

2 Materials and methods

An ingot composed of Al 65 at.%, Cu 15 at.% and Co 20 at.%, which refers to quasicrystalline materials obtained in the melt-spinning process [23], was prepared using metals of 99.9% purity in an induction furnace. Subsequently, fragments of the ingot were remelted and cast in the melt-spinning process, conducted under the following conditions: a helium pressure of 0.25 MPa, a quartz crucible nozzle diameter of 0.7 mm, and a linear velocity of the rotating wheel set to 20 m/s.

The microstructure and chemical composition of the melt-spun alloy were analysed using a scanning electron microscope (SEM) FEI E-SEM XL-30 equipped with an energy-dispersive X-ray spectrometer (EDS). The cross section of ribbons was examined in backscattered electrons mode. A detailed microstructure and phase composition study was performed using a transmission electron microscope (TEM) FEI Tecnai G2, which featured a high-angle annular dark-field scanning transmission electron microscopy detector (HAADF-STEM). Chemical composition mapping was carried out using a ThermoFisher Titan Themis G2 200 Probe Cs-Corrected transmission electron microscope equipped with a ChemiSTEM EDS system for chemical analysis. TEM observations were performed on lamellas prepared by the focus ion beam (FIB) method using a ThermoFisher Scios 2 Dual Beam microscope. Samples were cut from the surface of the ribbon.

To prepare material for catalytic tests, a vibration micro mill Fritsch Pulversiette 0 was used to pulverise the ribbons, followed by sieving with a Fritsch Analysette3 analyser to separate particles below 32 μm. The powder was also used to examine the phase composition of the alloy using a Bruker D8 Discover X-ray diffractometer (XRD) with CoKα radiation (1.7903 Å). For the direct comparison of obtained spectra with literature reports, the peak positions were recalculated to CuKα radiation (1.5406 Å).

Catalytic tests in the liquid environment were conducted for the phenylacetylene hydrogenation reaction, serving as a model reaction for the semihydrogenation of unsaturated hydrocarbons. Before the reaction, the catalyst powder was cleaned with a $NaBH_4$ aqueous solution until the end of hydrogen release. The cleaned catalyst was then directly transferred to an agitated batch glass reactor. The following reaction conditions were used: 50 mg of catalyst, 2-propanol as a solvent, 300 mol of phenylacetylene, a total reaction mixture volume of 75 ml, a temperature of 50 °C, an H_2 pressure of 5 bar, and a reaction time of 1 h. The composition of the reaction mixture was analysed using a Clarius 500, Perkin Elmer gas chromatograph with helium as a carrier gas. After the reaction, the recovered catalyst was examined in terms of evaluating its stability by X-ray diffraction, scanning and transmission electron microscopy methods.

3 Results and discussion

3.1 Material characterisation

The analysis of the X-ray diffractogram of the pulverised ribbon (Fig. 1) confirmed the presence of a decagonal

quasicrystalline phase (DQC) with reflection positions almost the same as for Al-Co, Al-Co–Cu and Al-Co–Ni quasicrystals [24]. The indexing of reflections referring to the DQC was carried out according to the work [25]. Additionally, low-intensity reflections were observed, and their position indicates the presence of Al_2Cu [26] and Al(Cu, Co) [16] phases that were reported in Al-Co–Cu alloys with similar compositions [27]. Reflections of the $Al_{13}Co_4$ approximant, often identified in alloys with similar chemical composition and containing quasicrystal [28], were not found.

Observations of the cross section of ribbons using a scanning electron microscope revealed uniform morphology across the thickness of the material, with the porosity occurring near the free surface (Fig. 2). The thickness of ribbons is in the range of 50–80 µm. EDS analysis obtained for the observed area of the ribbon indicates the chemical composition containing 66 at.% Al, 14 at.% Cu and 20 at.% Co. Higher-magnification observations of the near free surface area show multiphase microstructure with irregular grains surrounded by the lighter second phase (Fig. 2b). Occasionally, darker grains, referring to the phase with a higher share of aluminium, occur. Near the wheel side of the ribbon, the light areas of the second phase are arranged in vertical lines, suggesting columnar growth of the main phase in this region (Fig. 2c).

A detailed microstructural study of ribbons was conducted by the TEM techniques. Bright-field observations clearly show dendrites or cellular grains (Fig. 3a, c). Selected area diffraction patterns collected in the area of these grains reveals tenfold and twofold symmetry, which is typical for decagonal quasicrystals (Fig. 3b–c). Diffraction with twofold symmetry has a main periodic arrangement with a periodicity of 0.4 nm. Additional lines with lower intensity occurring between brighter reflection rows have a periodicity of 0.8 nm. These values suggest the presence of D_1 and D_2 quasicrystal variants. D_2 occurs in binary Al-Co rapidly solidified alloys with the composition near $Al_{13}Co_4$ phases [29]. In the areas between the DQC grains, two types of obtained diffraction patterns were identified as Al(Cu, Co) and Al_2Cu phase (Fig. 3e–f).

Additionally, EDS mapping was performed to determine phase distribution within the material (Fig. 4). The average composition of DQC grains was about 62 at.% Al, 23–26 at.% Co, 12–16 at.% Cu (points 1 and 7 marked in the HAADF-STEM images presented in Fig. 4a, d). The areas between quasicrystalline grains have diversified chemical composition, revealing varied aluminium concentrations, as shown in the line analysis in Fig. 4c. The results confirm the presence of two phases. Both of them are Co depleted (2–5 at.%) and enriched in Cu (38–47 at.%) compared to DQC (table in Fig. 4f). Based on electron diffraction patterns, the most frequently observed phase located between dendrites containing about 60% at. Al (points 2, 3 and 6 in Fig. 4a, d) was identified as Al_2Cu (Fig. 3e) with a tetragonal structure, while the phase with a lower Al content of about 50 at.% (points 4 and 5 Fig. 4a) as Al(Cu, Co) phase. The latter is a low-temperature phase with a B2 cubic structure, a ternary extension of the binary AlCo phase, which dissolves up to 45 at.% of Cu [30]. Congruous phase composition results were reported for alloys with similar chemical compositions but annealed at temperatures above 1050 °C [31]. According to the literature, the phase composition of Al-Cu-Co quasicrystal is sensitive to transition metals proportion. Rosas showed that for alloys containing Al, 20 at.% Co and Cu,

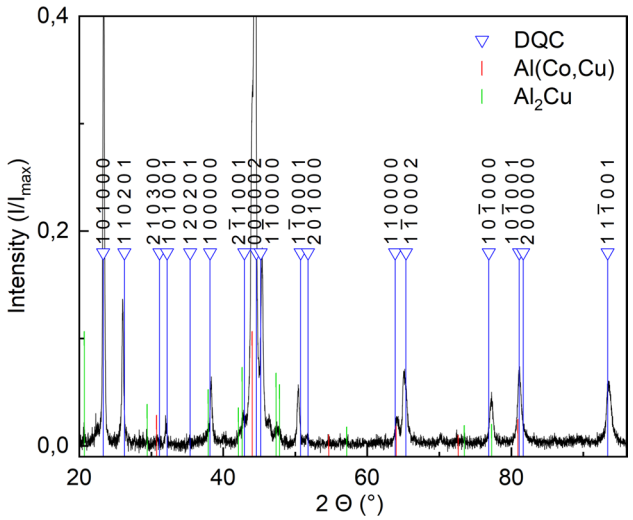


Fig. 1 X-ray diffractogram of the pulverised ribbon with marked reflection positions for decagonal quasicrystal [14], Al_2Cu [26] and Al(Cu, Co) phases [16]

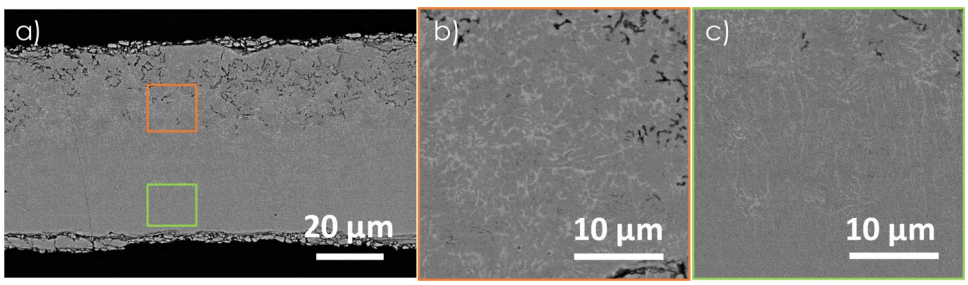


Fig. 2 SEM BSE microstructure of the cross section of the ribbon (**a**) with the magnified near free side region (**b**) and wheel side region (**c**)

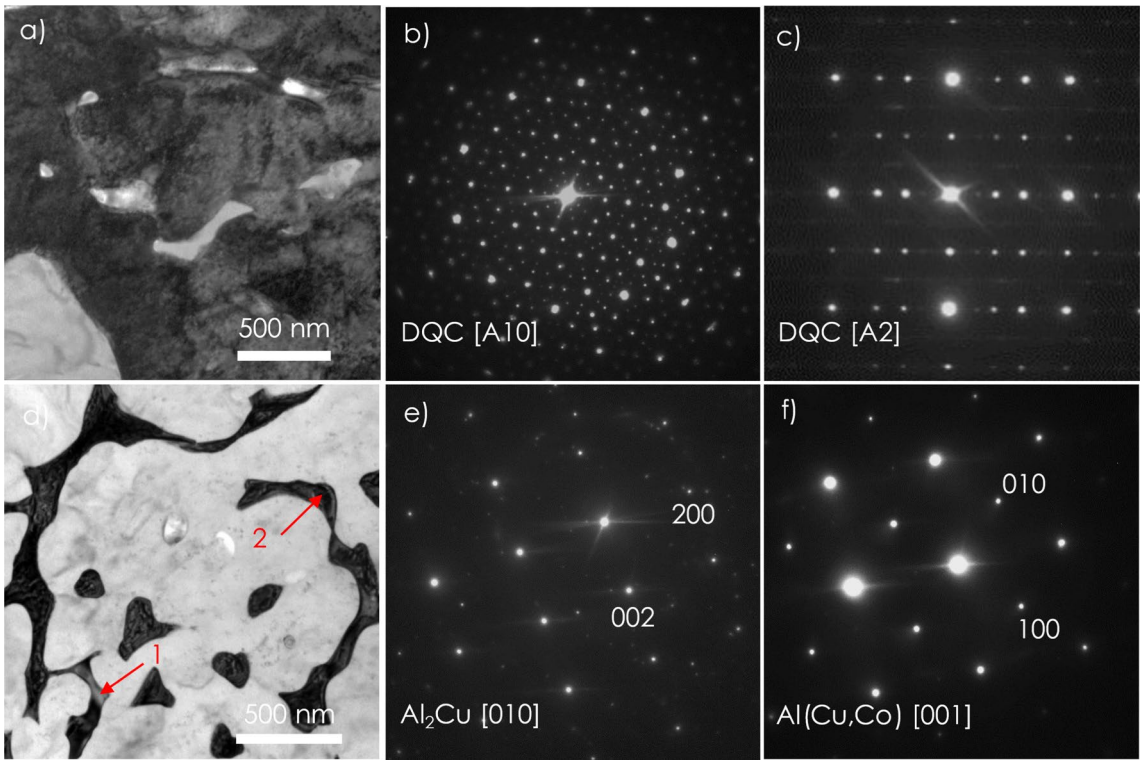
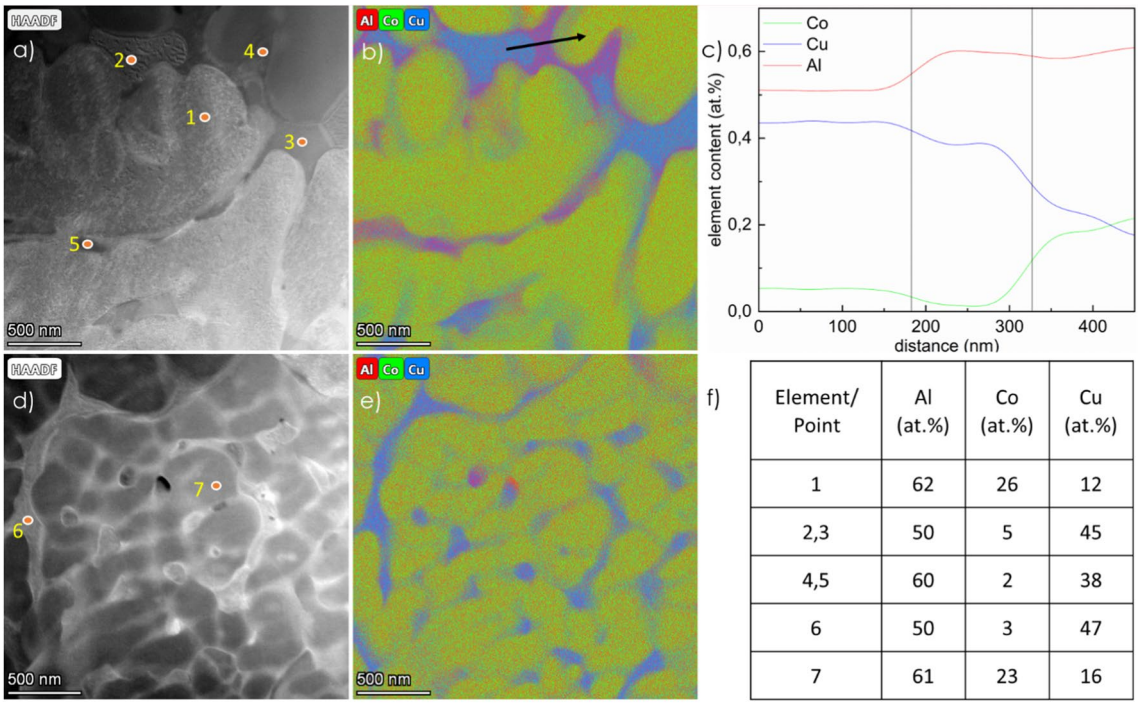


Fig. 3 TEM bright-field images of the selected areas of the ribbon (**a**, **d**) with electron diffraction patterns referring to decagonal quasicrystalline phase (**b**, **c**) and crystalline phases occurring in regions between quasicrystal grains (**e**, **f**)



Element/ Point	Al (at.%)	Co (at.%)	Cu (at.%)
1	62	26	12
2,3	50	5	45
4,5	60	2	38
6	50	3	47
7	61	23	16

Fig. 4 HAADF-STEM images of the selected areas of the ribbon (**a**, **d**) with EDS chemical composition maps (**b**, **e**), line analysis of the composition of the material in the area of Al_2Cu, Al(Cu, Co) and DQC phases presence (**c**), chemical composition of the material in the indicated points (**f**)

the single-phase alloy could be obtained. However, while increasing the Cu content at the expense of Co, the second cubic phase appears [19].

The literature reports indicate the possibility of homogenisation of Al-Cu-Co alloy with applied composition to obtain the single-phase decagonal quasicrystalline thin metal ribbons [32]. However, it would be beneficial to manufacture homogeneous alloy directly in the melt-spinning process by adjusting casting conditions.

3.2 Catalytic properties

The catalytic properties of the manufactured alloy were verified for the phenylacetylene hydrogenation reaction with the course presented in Fig. 5. The reaction requires the presence of hydrogen and a catalyst. The desired product of the reaction is styrene; however, under the reaction condition, the spontaneous subsequent formation of ethylbenzene occurs. The amount of ethylbenzene can be modified by the selection of the proper catalyst and adjustment of reaction parameters. The efficiency of the reaction can be adjusted by increasing the temperature of the mixture. The experiment was carried out under mild conditions, which may result in a lower fraction of the desired product compared to higher reaction conditions [9]. Nevertheless, this approach aims to develop processes with reduced energy costs.

The gas chromatography measurements of the chemical composition of the reaction mixture were taken every 10 min, and the results obtained are presented in Fig. 6. After 1 h, the phenylacetylene conversion was about 45%.

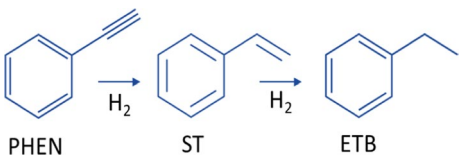


Fig. 5 Phenylacetylene hydrogenation reaction path in the presence of catalyst

However, further reaction could provide higher conversion of the substrate, as the reaction shows the selectivity to styrene, which achieved a value above 60% for the conversion in the 30–40% range. The results obtained can be directly compared with the performance of binary Al-Co alloy with the single-phase structure of a decagonal quasicrystal. This material was subjected to a phenylacetylene hydrogenation reaction with the same parameters applied [13]. Al-Co catalyst provided a substrate conversion of over 80%, but the efficiency was slightly lower for the multiphase alloy with the same chemical but complex phase composition. These results and the effect of the reaction with the Al-Cu-Co catalyst indicate the importance of phase stability for catalytic processes. Another interesting thing is the selectivity to styrene. In the mentioned work, during a 1 h reaction, the share of ethylbenzene was higher than styrene in every measurement point. Here, Al-Cu-Co alloy, despite the multiphase composition, provided a higher share of styrene, which indicates the legitimacy of further attempts to create a single-phase alloy and study its properties in catalysis. Recent reports indicate continuous development of this research direction other noble-metal-free intermetallic phases reveal excellent catalytic performance. For example, some works evaluate catalytic properties of Ni-Ga [33], Ni-Si [34] or ternary Ni–Cu–Sn [35] intermetallic phases in the form of nanoparticles in phenylacetylene hydrogenation reaction with high substrate conversion and selectivity. However, manufacturing of such materials is a complex process that requires the use of complex precursors, so searching for competitive alternatives is still reasonable.

3.3 Characterisation of recovered powder

After the reaction, the catalyst was rinsed in propanol, dried and examined in terms of phase stability. The X-ray diffractogram of recovered powder contains reflections in the same positions as for the initial material. No additional peaks were observed, which indicates the phase stability of the applied alloy. The similar intensity of individual reflections does

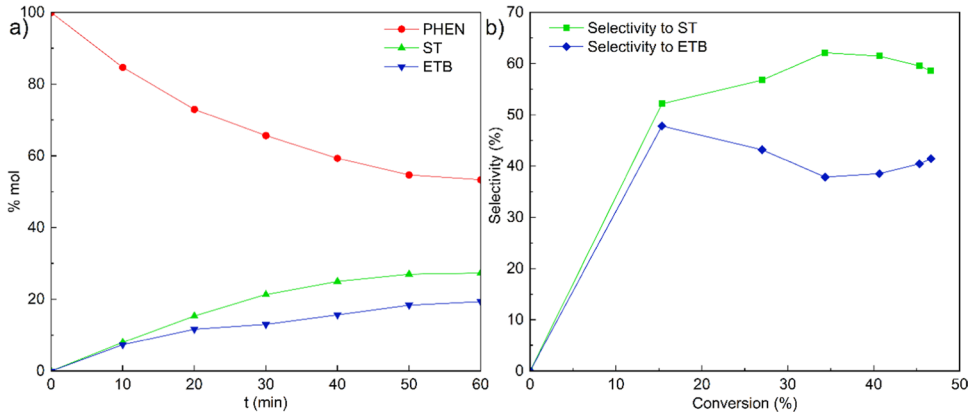


Fig. 6 Hydrogenation of phenylacetylene to styrene with Al-Co–Cu powder applied as catalyst (**a**) with selectivity to styrene and phenylacetylene in the reaction (**b**)

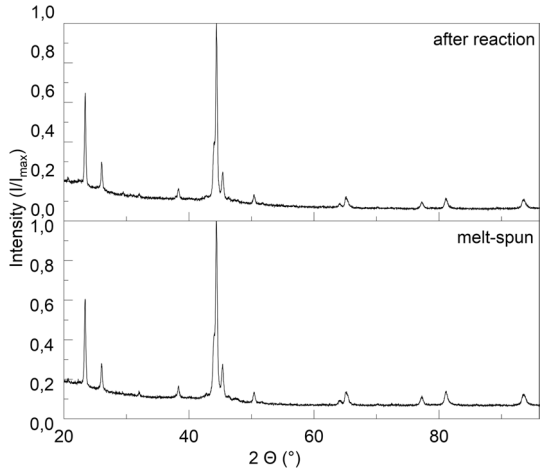


Fig. 7 X-ray diffractogram of the powder recovered after the reaction compared to the diffractogram collected for the melt-spun alloy

not indicate changes in the share of individual phases. This result was expected, considering the low temperature at which the reaction was carried out (Fig. 7).

SEM observations of the powder before and after the reaction do not reveal significant changes in the morphology (Fig. 8). The only difference which is visible on SEM images is the lower share of the smallest particles, which may be an effect of powder retention on filters during powder preparation for reaction and afterwards collection. The TEM observation of single particles does not indicate any visible surface degradation (Fig. 9). The small particles tend to agglomerate, but their sizes are still in the range of a few micrometres. The chemical composition of recovered powder evaluated by the point EDS analysis in the area near the particle edge revealed Al, Co and Cu, which are components of the alloy, and additionally O, C, and Ni. The presence of nickel and carbon results from the TEM grids used for the observations. The high share of oxygen may stem from surface oxidation, which may result from catalyst preparation, reaction or recovery procedure. These results indicate the high catalyst potential in the subsequent cycles of the reaction. The stability is the great advantage of intermetallics being applied as catalysts in mild-condition reactions. Similar phase stability was observed for the previously mentioned Al-Co catalyst; however, in that case, a build-up on the particles was visible. Moreover, on the XRD spectra, the board amorphous peak occurred. This powder contamination may be an effect of skipping the rinsing of the catalyst after

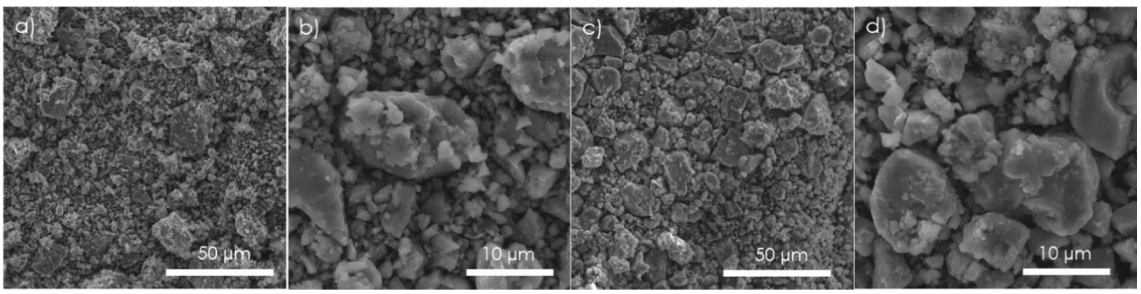


Fig. 8 The morphology of pulverised and sieved powder obtained from melt-spun ribbons before the reaction (**a**, **b**) and after reaction (**c**, **d**) observed in SEM SE

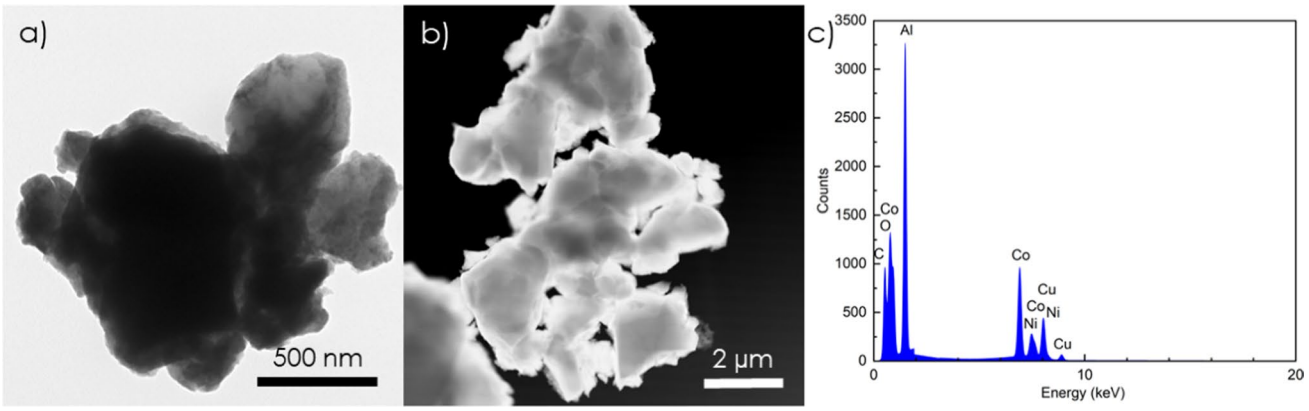


Fig. 9 TEM bright-field (**a**) and STEM (**b**) images of catalyst recovered after reaction with EDS spectrum (**c**) registered in the near-surface area

the reaction [13]. For the Al-Cu-Co catalyst discussed in the present work, cleaning after the reaction was applied, which provided the XRD diffractogram without artefacts.

4 Conclusions

The main goal of the presented work was to manufacture, characterise and verify the catalytic potential of the alloy with composition referring to the quasicrystalline phase from the Al-Cu-Co system. The experimental results obtained can be summarised as follows:

(1) The melt-spinning casting of $Al_{65}Co_{20}Cu_{15}$ alloy resulted in the formation of ribbons comprised of decagonal quasicrystalline aggregates surrounded by Al(Cu, Co) and Al_2Cu phases. Despite the broad range of composition for Al-Cu-Co quasicrystal and literature reports indicating the possibility of manufacturing quasicrystalline alloy with the composition used in rapid-solidification conditions, the obtained material was multiphase. Further casting optimisation in terms of wheel speed and chemical composition adjustment is required to produce material with the assumed phase composition.
(2) Pulverised ribbons exhibit catalytic performance in phenylacetylene hydrogenation reaction with substrate conversion of about 45% after 1 h and selectivity to styrene in the range of 60%, which encourages further material composition and reaction conditions. Additionally, for the catalytic activity improvement, the treatment providing catalyst specific surface area development, like dealloying of precursor alloy, could be applied.
(3) The catalyst persists to be stable under the reaction conditions. Recovered powder reveals the same phase composition as melt-spun material. The morphology of powder particles examined using SEM methods also does not change after the reaction. The obtained results suggest that the quasicrystalline structure remained unchanged after the reaction, which is a promising perspective for the reuse of such a group of catalysts.

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Data availability All data included in this work are available upon request from the corresponding author.

Declarations

Conflict of interest The authors have no competing interests to declare that are relevant to the content of this article.

Ethical approval This article does not contain any studies with human participants or animals performed by any of the authors.

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