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Development of Aluminum Scandium Alloys for Hydrogen Storage Valves

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For the development of aluminum scandium alloys to produce a hydrogen storage valves, various aluminum alloy series with different Sc and Zr contents are tested. The alloys are mainly characterized by hardness measurements and tensile tests. The hardening curves are recorded for different temperatures and compositions, and the selected parameters are validated with the corresponding mechanical tests. The alloys 5083 + 0.2% Sc and AlSi16Mg1Sc0.4 are selected for the production of hydrogen valves by forging and additive manufacturing.

1. Introduction

Lightweight materials such as aluminum-based alloys have long been developed and their performance optimized.[1] Therefore, the potential for significant improvement is generally low. A very effective method of improving the properties of aluminum (Al) alloys is the addition of small amounts of the element scandium (Sc).[2] The hardening potential of Sc in Al was first discovered in the 1960s. Scandium is the most effective element known for the microalloying of Al and its alloys.[3] Sc concentrations between 0.05 and 0.50 wt% are typical. There are three main effects that can be achieved by the addition of Sc to Al alloys: 1) grain refinement, 2) inhibition of grain boundary movement, and 3) hardening through the formation of Al_3Sc dispersoids during artificial aging.[3b] The improvement in strength is mainly caused by the formation of Al_3Sc nano-precipitates and amounts to around 50 MPa per 0.1 wt% Sc addition. These particles are formed by heat treatment at elevated temperatures, typically in the range of 200–400 °C, and are very finely distributed.[4] In addition to a higher strength, Sc has an antirecrystalization effect, improves toughness, weldability, corrosion properties, and allows superplasticity in Al alloys.[2,5] Al-Sc alloys have developed into a new generation of structural materials.[3b]

However, the high price of Sc, the scarcity of ores, a complicated extraction process, and the monopoly position of individual countries in its supply have so far prevented widespread use of Al-Sc alloys. In recent years, Canadian, Australian, and European initiatives have succeeded in opening up new deposits and developing new processes for Sc extraction. For example, what is believed to be the largest deposit of rare earths (including Sc) was recently discovered in Kiruna in northern Sweden.[6] This is expected to lead to a significant reduction in the current price of Sc, resulting in a wider range of applications.

The combination of Sc and zirconium (Zr) in Al-Sc alloys is particularly effective as it forms a core–shell structure of $Al_3(Sc, Zr)$ dispersoids, where Zr_3Sc is formed as a covering shell around the Al_3Sc dispersoids, preventing further growth and stabilizing them at high temperatures, such as those required in forging.[7] A similar hardening effect can be achieved by replacing 30–50% of the amount of Sc with Zr, which also has a positive economic effect, as the price for Zr is much lower than of Sc.

Hydrogen as an energy supplier is gaining interest over the past decades. Constant innovation and the need for materials development, especially in aviation, rail vehicles, shipping, and road transport, are required. Aluminum 6061 T6 and stainless steel (1.4435, 1.4475) are the materials currently used for hydrogen valves in the transport industry (see **Figure 1**).[8] Such valves are installed in hydrogen tanks for hydrogen-driven vehicles and designed for operation under pressures of up to 700 bar.[8] Using aluminum alloys reduces the weight compared to conventional steel solutions and therefore harbors the potential to reduce CO_2 emissions. A hydrogen valve is a complex component that requires a large number of special tools and long and precise machining times in production. In order to achieve the tolerances needed for hydrogen tightness, complex machining cuts are required, which currently make the valve a relatively expensive component in production. At the same time, light weight and high strength are not the only requirements for this type of valves, as good toughness and machinability as well as corrosion resistance and hydrogen compatibility and resistance against embrittlement at high pressures are required.

Improving the mechanical properties of 5xxx alloys to the level of 6xxx, but with the advantages of a 5xxx alloy such as superior corrosion resistance, better weldability, formability,

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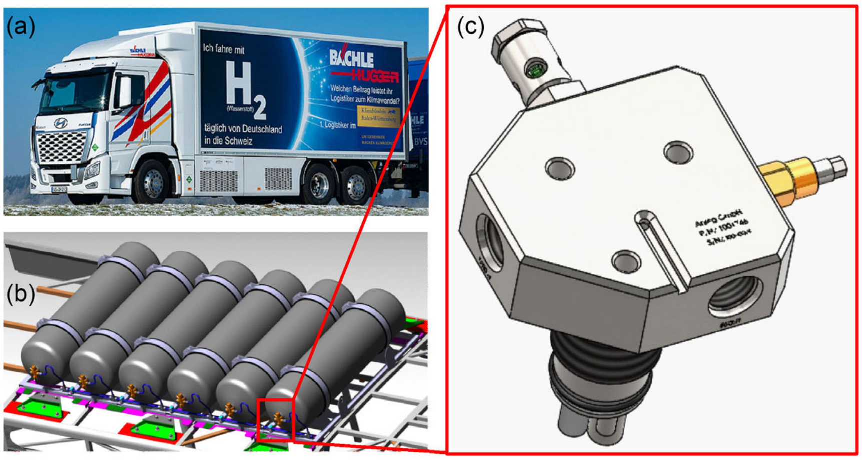

Figure 1. a) Hydrogen powered vehicle, b) hydrogen tanks with steel hydrogen valves, and c) close view of a standard steel hydrogen valve, reproduced with permission of Argo-Anleg.[8,15]

and toughness, offers a decisive advantage. A better formability leads directly to cost savings in the manufacturing process. The price development of Sc will be decisive for the economic use of Al-Sc alloys. An alloy with 0.2% Sc by weight requires 2 kg of Sc per tonne of Al. A kilogram of Sc currently costs more than $2,000 and is therefore comparable in price to a tonne of aluminum, which has a market price of around $2,700 per tonne,[9] while the price for a comparable and competitive alloy (AA 6061) costs around $3,000–3,500 per tonne. At the current price, the use of even the smallest quantities of Sc is therefore only economical for special applications with added value. The Canadian Sc supplier Rio Tinto has recently adopted a particularly cost-efficient Sc process route as by-product of titanium oxide production.[10] The price of the master alloy AlSc2 is currently $40 000 per tonne, resulting in a price of $4,000 per tonne for an alloy containing 0.2% Sc, which is around 17–33% more expensive than AA 6061. A possible future raw material supply chain and increased demand could drive prices down even further.

The aim of the work is first to perform a comprehensive screening study throughout different Al alloy series with Sc addition to figure out the best potential for properties improvement focused on commercial alloy compositions. Second, different alloys with the greatest potential are selected for optimization for production following forging and additive manufacturing production routes. For that purpose, a large number of alloy composition with and without Sc and Zr addition and different heat treatment temperatures and times are evaluated comparing their hardness and tensile strength. Further, preliminary designs and prototypes of a hydrogen valve are presented to prove their suitability for lightweight construction and hydrogen applications.

The alloy 5083 was selected for forging, and a more detailed optimization was performed. The latter was mainly achieved by optimizing the solution heat treatment and age hardening temperatures and times. Furthermore, the addition of Zr and the influence of mechanical deformation in the form of extrusion were investigated. Vickers and Brinell hardness were evaluated as the first quality criterion, while tensile tests were performed to validate the results. For additive manufacturing, the $\text{Al-Si}_x\text{-Mg}_y$ system was selected as basis, as AlSi10Mg is the most applied alloy for additive manufacturing at present.[11] The Si and Mg amounts were optimized and AlSi16Mg1 for this route.

2. Results and Discussion

2.1. Development of the Wrought Alloy

Various alloys from different aluminum alloy groups, namely 3xxx, 5xxx, 6xxx, and 7xxx, were evaluated to first perform a rough screening of their potential. The maximum effect of 0.05–0.2% Sc addition on the hardness is shown in **Figure 2**. We only noticed minimal changes in the hardenable high-performance 6xxx and 7xxx alloys, and even a negative trend in the 7xxx series. The reason for this is that Sc could not develop its full hardening effect in this series through artificial hardening, as the conventional heat treatment temperatures required for these series are

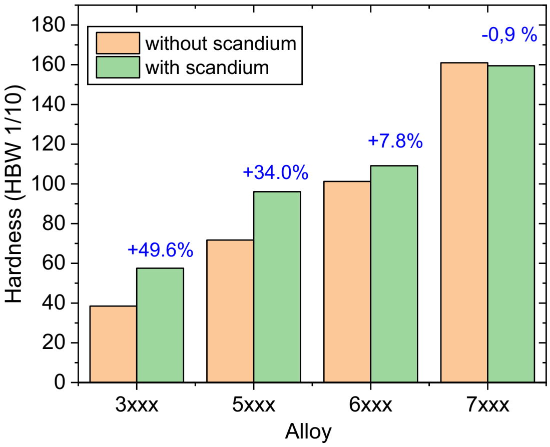

Figure 2. Maximum effect of 0.05–0.2% Sc addition on the hardness of cast 3xxx, 5xxx, 6xxx, and 7xxx alloys.

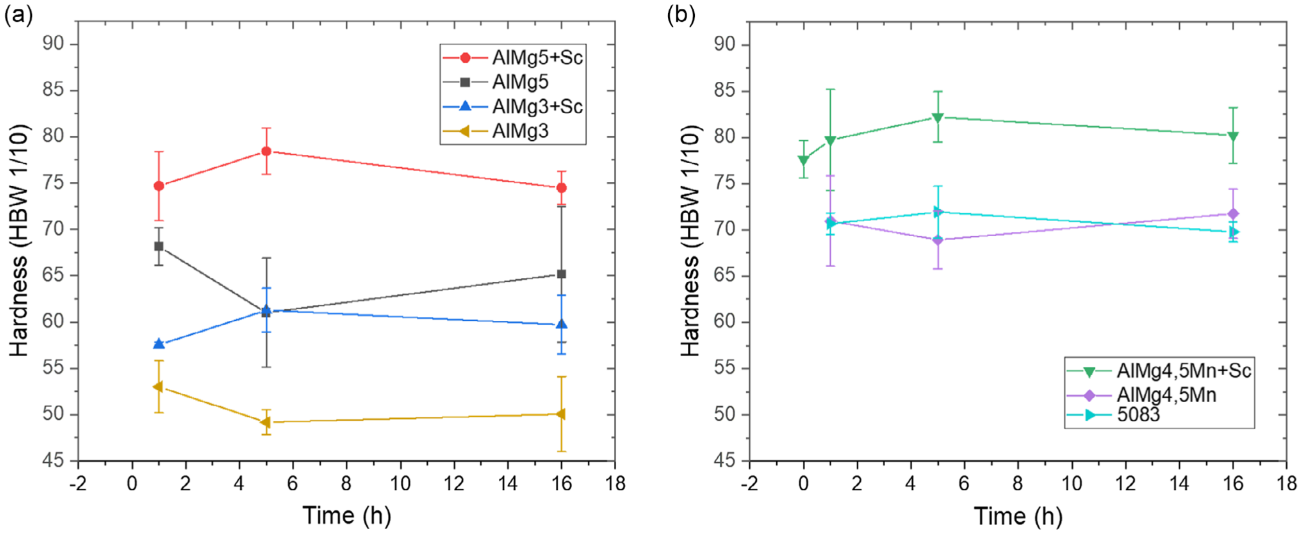

Figure 3. Hardness plotted over the heat treatment time for a) AlMg3, AlMg5, and b) AlMg4.5Mn with and without 0.2% Sc addition compared to commercial 5083. All samples were cast, water quenched, solutionized at 540 °C for 30 min, and then heat treated for 1, 5, and 16 h at 300 °C.

in the range of 90–240 °C instead of 250–350 °C as required for formation of the coherent Al_3Sc phase.[12] Particularly in alloys with a high silicon (Si) content, Sc and Si react to form different scandium silicides, so that less Sc is available for the desired Al_3Sc dispersoids. This detrimental effect on strength can be observed from a Si content of 0.4 %.[13,3b] On the other hand, a significant increase in hardness was found for the 3xxx and 5xxx alloys, up to 49.6% was found for a 3003 alloy and up to 34% for 5083. No heat treatment was required for the latter two series, which is why they could be optimized for the precipitation of Al_3Sc dispersoids.

From now on, we focused on various 5xxx alloys, which represent a good compromise between medium to high strength, good ductility, and excellent fatigue and corrosion resistance. With this in mind, the hardness development of various 5xxx alloys was measured for different curing times for AlMg3, AlMg5, AlMg4.5Mn with and without 0.2% Sc addition and the commercial 5083, see **Figure 3**. The results show that the greatest potential for improving the properties lies in AlMg4.5Mn + Sc. Therefore, the commercially available alloy 5083, which has a composition very close to that of AlMg4.5Mn, was selected as an industrially available alloy for further detailed investigation.

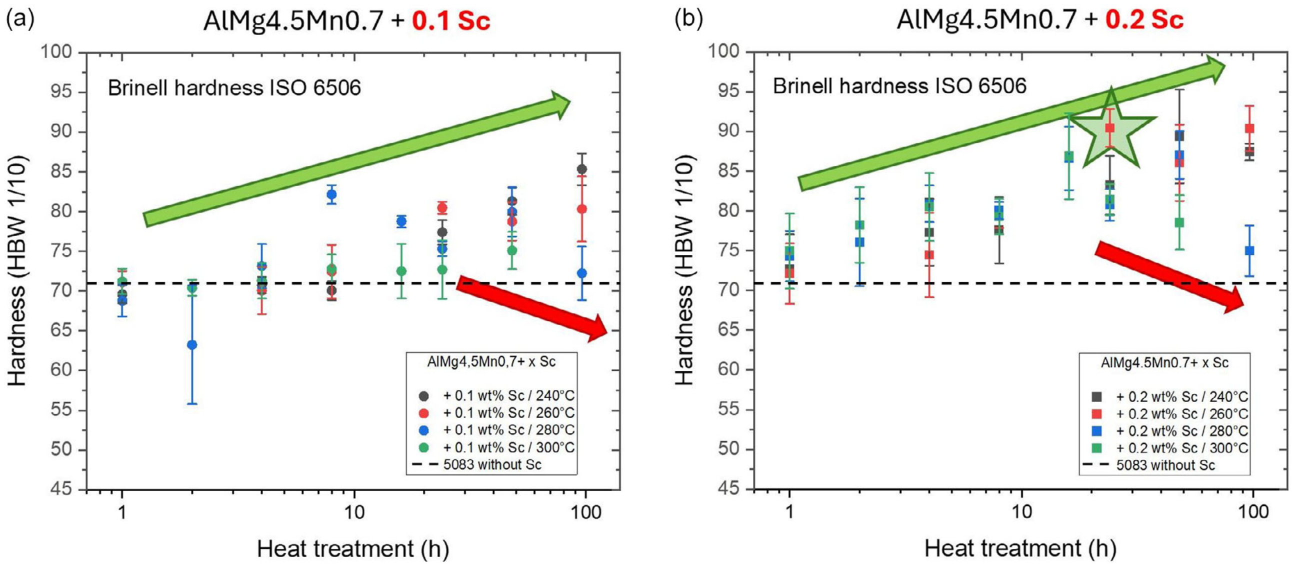

Figure 4. Hardness versus heat treatment time for different temperatures for the alloy AlMg4.5Mn0.7 + x Sc with a) 0.1% and b) 0.2% Sc addition. Hardness increases in most cases with time (green arrow), but decays under certain conditions for times longer than 24 h (red arrow). The hardness values are around 10% higher for 0.2% than for 0.1% Sc addition. The alloy 5083 + 0.2% Sc extruded at 450 °C, water quenched, and heat treated at 260 °C for 24 h was selected for the hydrogen valves (green star). Hardness of commercial 5083 without Sc is plotted as reference.

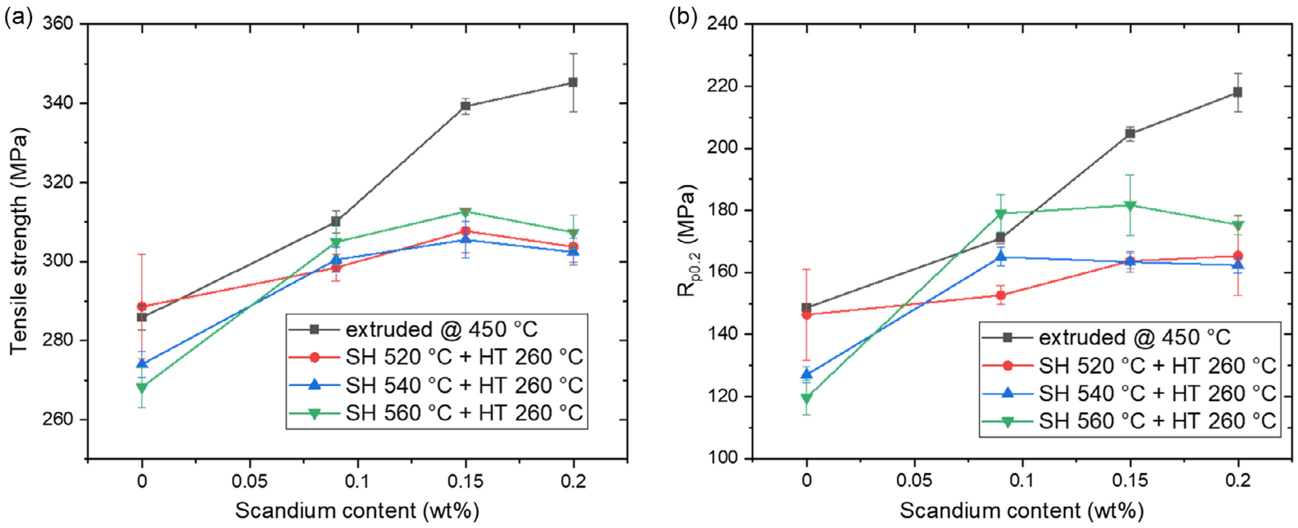

Figure 5. a) Tensile strength and b) yield strength $R_{p0.2}$ of 5083 + 0.2% Sc in dependence of the Sc content on AlMg4.5Mn0.7 + 0.2% Sc for different conditions: Only extruded at 450 °C and extruded at 450 °C and additionally solution heat treated (SH) at 520, 540, and 560 °C for 2 h followed by a heat treatment (HT) at 260 °C for 24 h.

In a further step, the amount of Sc and the heat treatment temperature and time were varied for alloy AlMg4.5Mn0.7 + x Sc, a composition close to the commercial alloy 5083. This alloy is renowned for its high strength, exceptional corrosion resistance, and excellent weldability. These properties make it ideal for high-pressure applications such as pressure vessels and cryogenic storage tanks. Notably, 5083 maintains its strength even after welding and is commonly used in shipbuilding and vehicle armor.

Samples of the composition AlMg4.5Mn 0.7 + x% Sc were cast, water quenched, solutionized at 540 °C, and heat treated. The amount of Sc (0.1% and 0.2%) and the annealing time (up to 100 h) and temperature (240, 260, 280, and 300 °C) were optimized. For this purpose, the dependence of the hardness on temperature and time was evaluated, as shown in **Figure 4**. We can observe that the absolute hardness values increase with heating time and are around 10% higher for 0.2% than for 0.1% Sc addition. Hardness increases in most cases with time, see green arrows in Figure 4, but decays for 280 and 300 °C heating temperatures for times longer than 24 h as indicated by red arrows in Figure 4. The best hardness values of up to 90 HBW could be achieved by AlMg4.5Mn0.7 + 0.2% Sc heat treated at 260 °C for 24 h, green star in Figure 4.

In the next step, the tensile and the yield strength at 0.2% elongation ($R_{p0.2}$) were measured for extruded samples at 450 °C with a precipitation heat treatment at 260 °C for 24 h, see **Figure 5**. The results were compared with samples, which were additionally solution heat treated at 520, 540, and 560 °C for 2 h before the extrusion with the aim to improve Sc dissolution. The results show that the highest tensile strength (Figure 5a) and the highest yield strength (Figure 5b) of with 345 and 218 MPa, respectively, were obtained for the specimens with 0.2% Sc addition without soluzioning heat treatment. The latter seems to bind some Sc that is later no longer available. Therefore, no solution treatment leads to the best results, as the extrusion step should take over the intended homogenization effect of a solution heat treatment, but allows the maximum reinforcement potential of the Sc.

2.2. Development of the Alloy for Additive Manufacturing

Laser powder sintering of the previously selected 5083 + 0.2% Sc alloy was performed for different Sc contents. Additionally, samples of another composition Al-Six-Mgy were produced by laser sintering of the elemental powers and master alloys for $x = 3$–20 wt%, $y = 0$–1 wt% with Sc and Zr additions as described in the experimental section, thus emulating the laser bed fusion process (LPBF). The Vickers hardness of the sintered specimens was evaluated in dependence of the Si content, see **Figure 6**. As expected, the hardness increases with increasing Si content. An average hardness of 70 HV can be observed for the commercially available, well established and easy

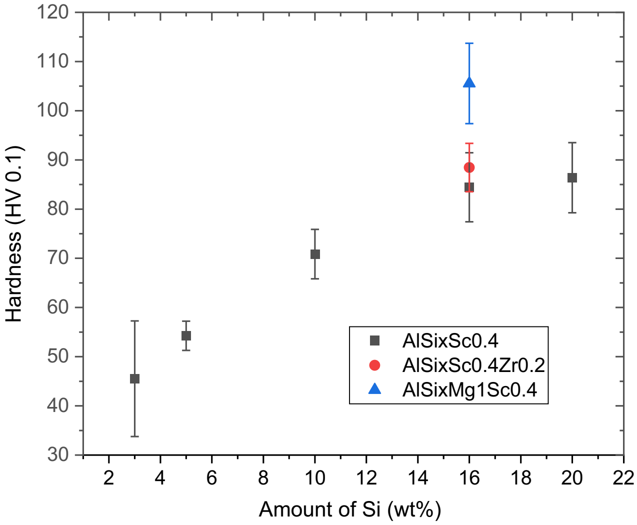

Figure 6. Effect of amount of Si and different Sc and Zr additions on the hardness of Al-Six-Mgy ($x = 3$–20 wt%, $y = 0$–1 wt%).

printable AlSi10 alloy, with 0.4% addition of Sc. The hypereutectic alloy AlSi16Mg1Sc0.4 delivered best hardness values of 105 HV and was selected for LPBF of the hydrogen valves.

Figure 7 shows optical images of the microstructure of AlSixSc0.4Zr0.2 for $x = 5$, 10, 16, and 20 wt%. The amount of eutectic phase increases with the Si content as expected. For the hypereutektik compositions ($x = 16$, 20), primary Si particles are found, correlating with the increase in the hardness. Despite small voids, the microstructure of such laser-sintered single powder layers is quite homogeneous.

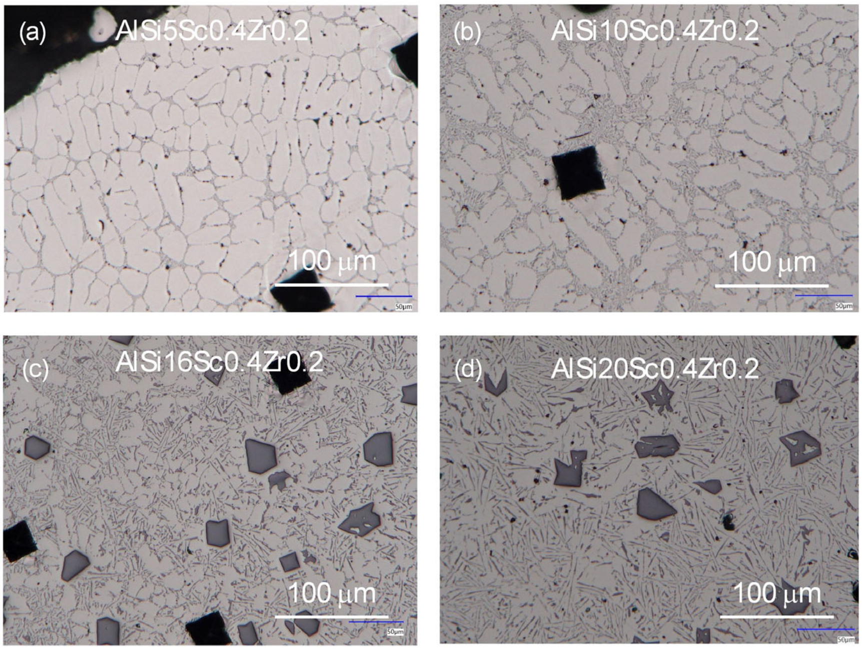

Figure 7. Optical images of the microstructure of AlSixSc0.4Zr0.2 for x = a) 5, b) 10, c) 16, and d) 20 wt%. Dark squares show Vickers hardness pyramid impressions.

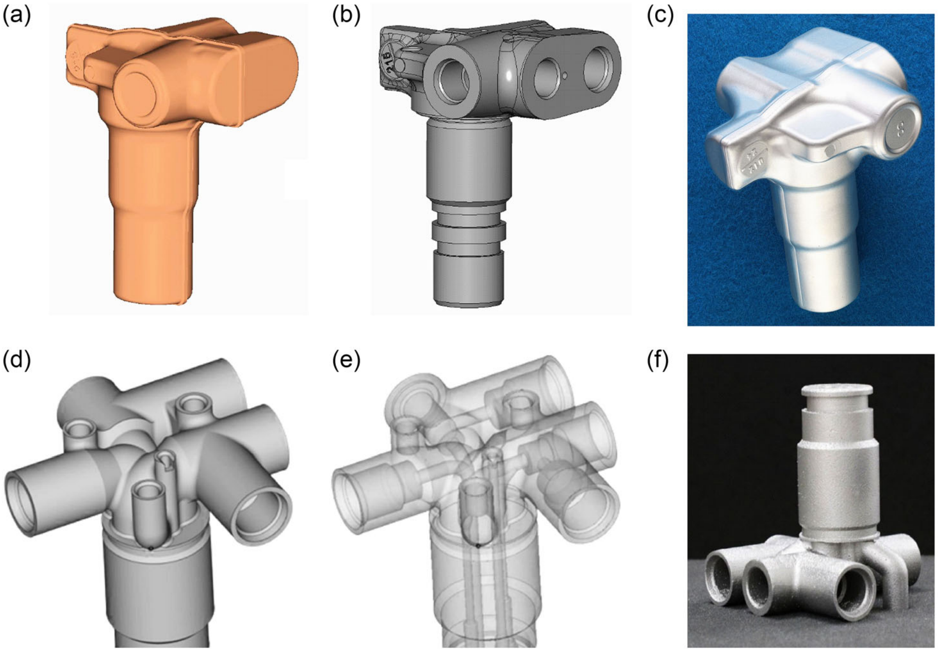

Figure 8. Hydrogen valves a) simulation of the forged billet, b) CAD design the further processed steps, c) blank of the forged billet, d) CAD design of the AM valve, e) detailed view of the inner conducts, and f) blank of the AM valve. Courtesy of LEIBER Group GmbH, Germany and Rosswag GmbH, Germany.

2.3. Hydrogen Valves

Hydrogen valves blanks were manufactured by forging and LPBF by LEIBER Group GmbH, Germany and Rosswag GmbH, Germany, respectively. The alloy 5083 + 0.2% Sc alloy, extruded and heated treated at 260 °C for 24 h, was selected for the forged hydrogen valve. Preliminary design and first production tests of the valve blanks can be observed in **Figure 8**. A hydrogen valve made by LPBF was also designed and additively manufactured by Rosswag GmbH with alloy AlSi16Mg1Sc0.4. The advantage of this process is a more near shape production than with forging, including prefabricated cavities and gas lines that enable easier post-processing. Another advantage is that no mold is required, but in contrast to forging, processing times are longer. LPBF production is more suitable for small series, while forging for larger serial production.

3. Conclusions and Outlook

After a comprehensive screening over different aluminum series to estimate the potential of scandium addition on hardness, the alloy composition and manufacturing parameters of different Al-based alloys with Sc additions were varied and optimized to produce a hydrogen valve by evaluating the hardness and tensile properties of the test specimens. Alloy 5083 + 0.2% Sc extruded and heated treated at 260 °C for 24 h was selected as the best choice for forging and AlSi16Mg1Sc0.4 for additive manufacturing. Extruded rods and powders from the selected alloys could be produced in commercially relevant dimensions. Hydrogen valve designs for forging and additive manufacturing were carried out and valve blanks were successfully produced. The next step will involve microstructural characterization, the investigation of material processing, corrosion and hydrogen compatibility tests, and finally the production of a ready-to-use hydrogen valve.

4. Experimental Section

The alloy was developed on a laboratory scale using small quantities of casting material to save costs. The next step toward upscaling was to pulverize the alloy in large quantities, using one half of the material for AM and the other for forging.

Casting, Extrusion and Characterization: Various Al-based alloys with different contents of up to 0.3% Sc and Zr were cast for alloy development. For this purpose, pure Al and Si as well as various master alloys were melted in a vertical furnace at 750 °C. The AlSc2 (in wt%) master alloy, provided by Rio Tinto, Canada, was cut into vertical precursor slices of the desired weight to avoid possible variations in Sc content, e.g., due to sedimentation effects of Sc by its fabrication. The alloying elements of the desired alloy were weighted, melted in a vertical furnace under air, and cast in a cylindrical graphite mold into ingots with a diameter of 29 mm. The ingots were then extruded into $20 \times 5\,\text{mm}^2$ and 500 mm long strips to reduce casting defects. In addition, an optional dissolution step was carried out to achieve a homogeneous distribution of the Sc atoms in the matrix before various heat treatments were finally carried out. The samples were grinded and polished for optical microscopy characterization and hardness measurements. Further, dog bone specimens were milled for tensile strength testing.

Metal Powder Production: The company Nanoval, Berlin, Germany, was commissioned by the project partner Gränges to atomize the selected alloys. Each batch produced 100 kg of alloy powder, of which around 50% was sieved into the desired size range for additive manufacturing and the rest industrially extruded into rods for forging.

Additive Manufacturing: Aluminum alloy strips were produced from the selected metal powder compositions using additive manufacturing. For this purpose, a portable single-mode fiber laser system (600 W max. power, 1070 nm wavelength) including a system for rapid beam control (“removeldFLEX” welding optics) provided by the Fraunhofer-Institute for Material and Beam Technology, IWS in Dresden was used and is further described in the literature.[14] Applying a laser power of 37 W, a spot size of 33 μm diameter, and a linear motion of $1.2\,\text{m min}^{-1}$, strips of 1 mm thickness and 40 mm length were produced for analysis. A slight stream of nitrogen ($10\,\text{L min}^{-1}$) as a shielding gas was blown over the sample to create uniform conditions and reduce oxidation.

Industrial Extrusion: To forge the valve blank, metal powder was compacted under hot isostatic pressure into solid green billets, which were extruded into bars with a diameter of 75 mm by Erbslöh Aluminium GmbH, Velbert, Germany.

Hardness Measurements: The hardness was measured with a Qness indenter, model 60 M, the samples for additive manufacturing with Vickers with a maximum test force of 0.98 N, and all other samples with Brinell with an indenter of 1 mm diameter and a maximum test force of 98 N according to DIN EN ISO 6506-1:2015-02. Hardness was used as a general benchmark and first-quality criterion for the alloy’s mechanical performance, while tensile tests were performed on selected samples to verify the results.

Tensile Tests: The tensile tests were performed on a Zwick/Roell Retroline Z100 testing device in accordance with DIN EN ISO 6892-1. TestExpert III Version 1.2 was used to record and analyze the data to obtain the tensile strength and the yield strength at 0.2% elongation $R_{p0.2}$.

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Conflict of Interest

The authors declare no conflict of interest.

Author Contributions

Francisco García-Moreno: data curation (equal); funding acquisition (equal); investigation (lead); methodology (equal); supervision (lead); validation (lead); writing—original draft (lead); (lead); and writing—review and editing (lead). **Tillmann Robert Neu**: formal analysis (lead); investigation (equal); methodology (equal); and writing—review and editing (equal). **Markus Eberl**: formal analysis (equal); investigation (supporting); methodology (equal); and writing—review and editing (supporting). **Paul Hans Kamm**: formal analysis (lead); investigation (equal); methodology (equal); and writing—review and editing (supporting). **Hans-Wolfgang Seeliger**: conceptualization (equal); data curation (equal); formal analysis (equal); funding acquisition (lead); supervision (lead); validation (lead); writing—original draft (supporting); and writing—review and editing (equal).

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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