

## Quasi-Isothermal Forging of Steel-Encapsulated Ti-6Al-4V Billets

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**Abstract.** Isothermal forging is a common method for manufacturing titanium alloys, but it involves complex processes and equipment. The oxidation of titanium leads to the formation of an alpha-case, which in turn promotes increased crack formation. To prevent this, inert gas is typically required. However, by encapsulating the titanium billet (Ti-6Al-4V) in a steel casing made of AISI 316L, a quasi-isothermal process can be achieved without the need for inert gas. This method maintains protection against oxidation while simultaneously reducing cooling. The sealing of the capsules is crucial to ensure that the titanium is effectively enclosed and protected from the surrounding gases. In this study, various encapsulation methods are compared, including rotary friction welding, diffusion bonding, and press-fitting a lid with an interference fit. The investigation involves differing contact conditions between the titanium and steel sleeve, as well as steel wall thicknesses of 2 mm and 4 mm. These factors showed no impact on the material flow or microstructure of the formed components. Encapsulation can prevent the formation of an alpha-case. Intermetallics form between the titanium and the steel capsule, depending on the contact conditions. The use of graphite as a separating agent prevents the formation of them.

### Introduction

Titanium and its alloys find numerous applications in lightweight construction, primarily due to their high specific strength-to-density ratio, with 60% of the density of steel, yet with similar strength [1]. Nevertheless, conventional machining of these alloys is challenging due to high tool wear and insufficient recycling processes for chips, posing ecological and economic issues [2, 3]. Forging offers a viable solution for producing near-net-shape titanium components. This method reduces material waste and allows precise microstructural control through temperature treatment, enabling the adaption of mechanical properties for specific applications.

Forging titanium alloys presents challenges due to their high reactivity with ambient gases. At elevated temperatures, titanium reacts particularly with oxygen [4]. This reaction leads to the formation of a brittle alpha-case near the surface. The alpha-case consists of a layer enriched with the  $\alpha$ -phase due to the alpha-stabilising effect of the oxygen, which can compromise the material's integrity [5]. Hence, reducing its formation is essential during processing. For instance, protective gases such as argon can be used to mitigate this issue. However, this involves increased demands on the peripheral process technology.

The titanium alloy Ti-6Al-4V alloy, which is an ( $\alpha + \beta$ )-alloy, undergoes a phase transformation at around  $T_{\beta} = 995 \text{ }^{\circ}\text{C}$  [2]. The hexagonal close-packed  $\alpha$ -phase is more brittle compared to the more ductile body-centred cubic  $\beta$ -phase. In conventional forging, the contact between the billet and die leads to cooling and potential phase transformation. The forming temperature is crucial as it affects the yield stress and attainable true plastic strain. The cooling of the workpiece surface due to the contact with the die results in severe deformation inhomogeneities [6]. To precisely control the forming temperature and distribution, allowing forging above the  $\beta$ -transus-temperature  $T_{\beta}$ , isothermal forging is used. This method aligns the die temperature with the billet, ensuring consistent temperature distribution. The die materials used in isothermal processes must withstand significant thermomechanical stresses. Nickel and molybdenum-based alloys are often employed for this

purpose, but they require the use of protective gases to prevent sublimation losses [7]. Overall, isothermal forging results in more complex equipment and additional energy for precise temperature control of the dies.

Encapsulation of titanium aims to prevent its reaction with oxygen. It also serves as a heat storage mechanism, which slows titanium's cooling process. Various approaches have been explored. Zhang et al. used steel foils around a TiAl billet with a thickness of 0.1 mm [8]. This method reduces temperature loss during upsetting. However, the removal of these foils is not discussed. Forged components with more complex geometries can present process difficulties. A mandrel would likely tear the foil, allowing oxygen to react with the titanium. As a result, this approach is less suitable for more complex geometries. In a direct powder forging process by Careau et al. Ti-6Al-4V powder is encapsulated in 1.3 mm thick 316L stainless steel. This encapsulation cracked, causing oxidation of the titanium surface [9]. They describe the easy decapsulation due to the intermetallics between titanium and the steel capsule. The brittle intermetallic phases crack during the second forging step. Excess material in the flash region is then cut, so that the stainless steel casing can be removed [9].

### **Preliminary Research.**

Previous studies focused on forming Ti-6Al-4V encapsulated in stainless steel with a capsule thickness of 4 mm [10]. The capsule, as detailed in Siring et al., serves to maintain the precise temperature conditions necessary for titanium forming, while preventing interaction with reactive atmospheric gases [11]. This approach facilitates a quasi-isothermal forming process, which eliminates the intricate procedural and technological demands typically associated with fully isothermal methods. Consequently, the encapsulation technique significantly optimises material efficiency relative to conventional machining techniques and also reduces the extent of subsequent machining processes required. Graphite proved effective as it enhanced material flow [10]. However, the capsule design did not completely prevent oxygen from reaching the titanium because the steel partially thinned out and ruptured. In the area between the fully pressed-in lid and the steel casing, a separation occurred. During the forming process, titanium flowed into this unprotected area and became oxidised. In a subsequent study, die geometries were numerically designed to produce the identical titanium core with different steel wall encapsulations of 2 mm and 4 mm despite varying material flow of the capsule [12]. In the experimental validation cracks appeared in the 2 mm steel capsule. The extent to which this leads to a reaction between titanium and oxygen has not yet been investigated.

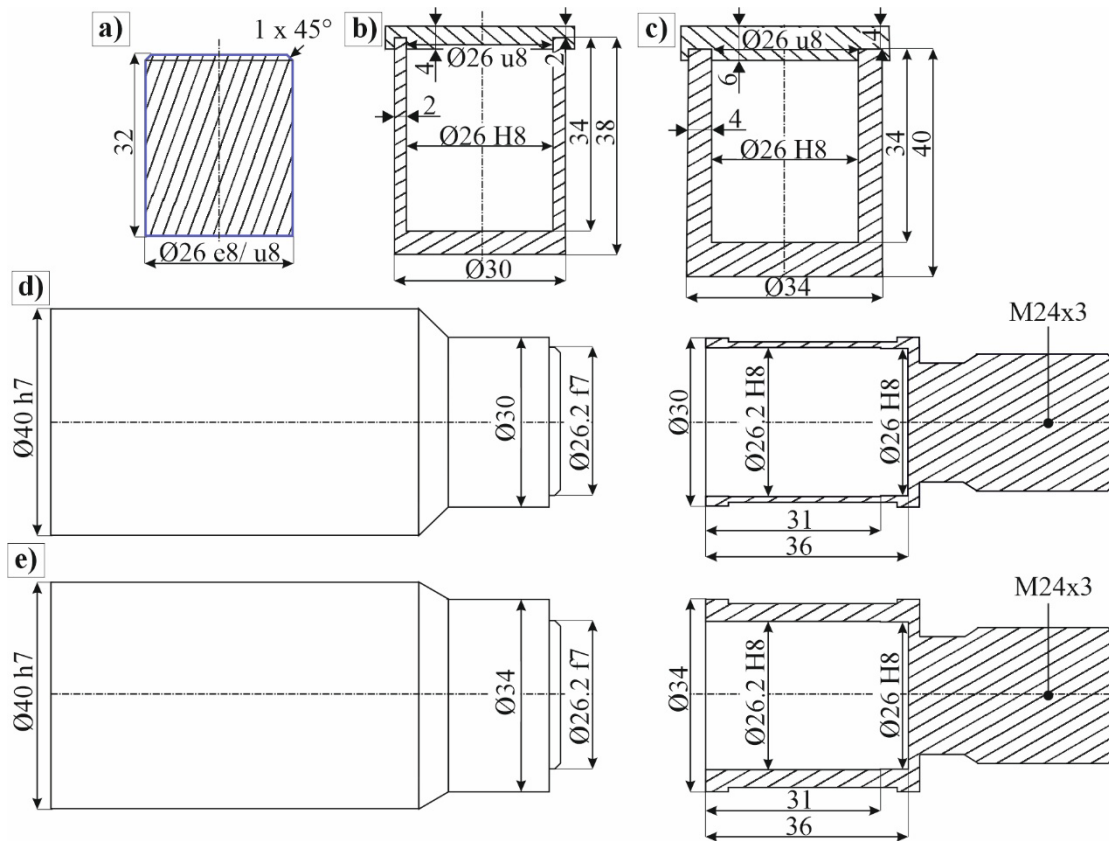
### **Research Gap.**

There is a research gap concerning the effects of encapsulation wall thickness and joining methods on the deformation behaviour and microstructure of titanium components. Specifically, it is not well-understood how different wall thicknesses (2 mm and 4 mm) affect material flow and microstructure, potentially leading to defects. Additionally, the effectiveness of various capsule sealing methods in preventing unintended openings and protecting the titanium core from oxygen exposure, to avoid the formation of an alpha case, requires investigation. Furthermore, the formation of intermetallic compound (IMC) at the interface between the titanium billet and the steel capsule is also not well understood during this process.

The hypotheses of this study propose that the steel capsule's wall thickness significantly impacts the deformation behaviour of titanium. Moreover, the effectiveness of different sealing methods, such as diffusion bonding, friction welding, and press fitting, in preventing oxidation will be assessed. Additionally, the formation of IMCs will be examined based on the contact conditions between the titanium billet and the steel capsule. Since the hypothesis is that the decapsulation of the titanium core is significantly influenced by the IMC understanding this is essential for subsequent work. To investigate the effects of the encapsulation strategy (including diffusion bonding, rotary friction welding, and press fitting) and capsule wall thickness on the microstructure, microscopic examinations will be conducted on the encapsulated titanium components. Following the forging trials, the components will be sectioned, undergo microscopic analysis, and be characterised using hardness measurements.

## Materials and Methods

The capsules are fabricated from AISI 316L stainless steel, with the dimensions of the components visible in Fig 1. Capsule wall thicknesses of 2 mm and 4 mm have already been utilised in previous studies; however, the microstructure and the formation of an alpha case were not investigated [12]. The illustration presents the titanium semi-finished product from TiA-6Al-4V with an interference fit and a clearance fit (Fig. 1a), capsules with a wall thickness of 2 mm (Fig. 1b) and 4 mm (Fig. 1c), and the semi-finished products for rotary friction welding with a wall thickness of 2 mm (Fig. 1d) and 4 mm (Fig. 1e). All components were manufactured using machining. The capsule is designed to be thicker at the upper and lower sections to increase thermal capacity and therefore reduce temperature loss of the titanium core. These areas experience the longest contact times with the dies and are subject to high mechanical stress due to the mandrel.



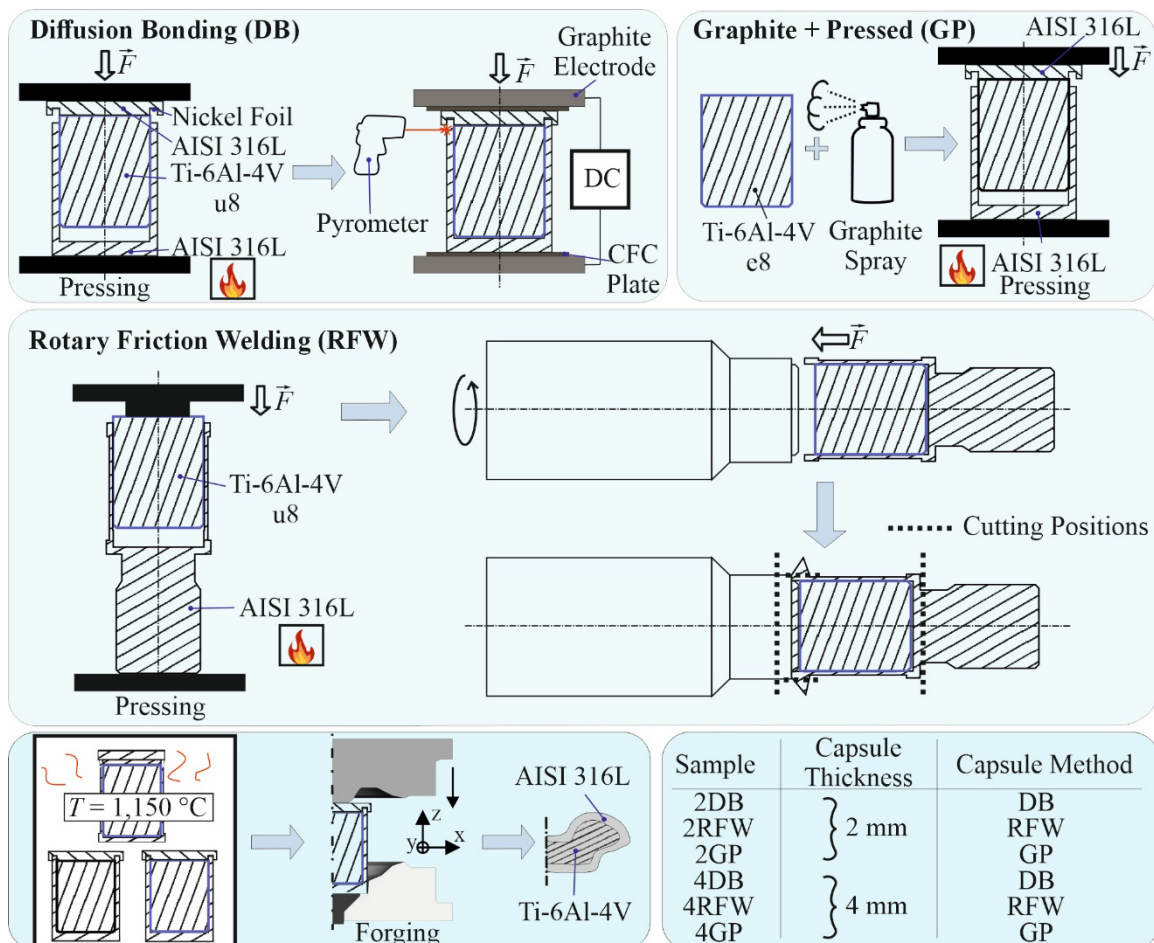
**Fig. 1.** Dimensions of the semi-finished products used: Ti-6Al-4V semi-finished product with an interference fit and a clearance fit (a), capsules with a wall thickness of 2 mm (b) and 4 mm (c), and the semi-finished products for friction welding with a wall thickness of 2 mm (d) and 4 mm (e).

Both the wall thickness and the joining technique of the steel capsules are varied while keeping the dimensions of the titanium core constant. Regarding the diffusion bonding process (DB), An et al. demonstrated that the joining of AISI 316L can be improved by using a nickel foil [13]. Accordingly, a nickel foil (h+s Präzisionsfolien GmbH, Vohenstrauß, Germany) with a thickness of 0.1 mm and a purity of over 99.2% is placed in the lid groove. The capsules were preheated to 300 °C to enable insertion of the titanium core and the lid, both of which have an interference fit after cooling. This process is carried out using a hydraulic hand press PJ20H (AC Hydraulic a/S, Viborg, Denmark). Due to the existing fit between the lid and the capsule, an airtight seal of the titanium core is achieved. The DB process then takes place using the sinter press DSP 507 (Dr. Fritsch, Fellbach, Germany). The machine heats the material using direct electrical current (DC) through a process known as Joule heating. During the process, the temperature is measured with a METIS M3 pyrometer (SensorTherm GmbH, Steinbach, Germany) and used to regulate and control the temperature. The set emission coefficient is 0.66. The temperature measurement takes place just below the lid, as this position reflects the temperature relevant for the bonding process (see Fig. 2).

The pyrometer is positioned using a built-in laser that corresponds to the measurement point. A low vacuum (20 mbar ambient pressure) is maintained during diffusion bonding to reduce oxidation of the capsule. In preliminary tests, deformation of the capsule and the titanium core occurred at the minimum adjustable pressure of 73.8 MPa at temperatures exceeding 700 °C. Since the diffusion process is temperature-controlled, the temperature is set to a maximum of 700 °C to prevent deformation of the semi-finished products while maximising diffusion. The heating rate is 40 K/min and the holding time at the bonding temperature is 1 h.

For the encapsulation of the titanium semi-finished product via rotary friction welding (RFW), the titanium semi-finished product is first pressed into the steel capsule as previously described. Between the titanium billet and the capsule, there is a gap above the lower section, which has an interference fit to ensure the fixation of the titanium within the capsule during the welding. After the insertion of the titanium, the capsule is then sealed with the lid using the friction welding machine Genius Plus R30 (KUKA, Augsburg, Germany). The steel lid component is secured to the rotating spindle side using three clamping jaws. The capsule, with the titanium pressed inside, is clamped on the translating slide side. The friction parameters were previously determined iteratively to ensure bonding between the capsule shell and the lid. At the start of the process, the still-open capsule is pressed against the lid rotating at 3,000 rpm with a force of 30 kN during the friction phase. Following the completion of the 2 mm friction path, the upsetting phase begins, in which the rotation is stopped and an upsetting force of 46 kN is applied for 5 s. After the samples have cooled, they are cut to size and the flash is removed (see Fig 2).

For coating the titanium core, the separating agent graphite SC 42 B (Dr. Fritsch, Fellbach, Germany) is applied. Then, a waiting period of 5 min is set to allow the solvent to evaporate. Following this, the titanium core is pressed in again as previously described. The resulting sample is named GP.



**Fig. 2.** Plan and procedure for different encapsulation approaches and forging experiments.

After encapsulating the capsulated titanium billets are heated for 1 h at 1,150°C in a convection oven to ensure the temperature is uniformly reached across the entire cross-section of the samples. The hydraulic press Schirmer + Plate is used for the forging experiments. The dies made from X38CrMoV5-3 (AISI H11) were designed in a prior study for the different wall thicknesses to produce the same titanium core [12]. For this purpose, the flash gap after numerical design for the capsule with a 2 mm wall thickness should accordingly be 2 mm, and for the 4 mm thick steel capsule, 3 mm. The forming speed is set at 30 mm/s for all samples. This speed is used to ensure minimal cooling of the titanium core [12].

For each variation of the experiment, five parts are forged. For further examination, one sample from each is cut using a wet abrasive cutter. The microstructure and material flow are examined using light microscopy on cross-sections etched with Kroll's reagent for 45 s. Subsequently, the samples were treated for 90 s in a V2A pickling bath at 65 °C. Microhardness tests are conducted at HV 0.05 in accordance with DIN EN ISO 6507-1 with QNESS 10A+ (QATM, Mammelzen, Germany) [14]. To achieve a dense positioning of measurement points along a line while ensuring the distances between the hardness indentations, the measurement points are staggered in their arrangement.

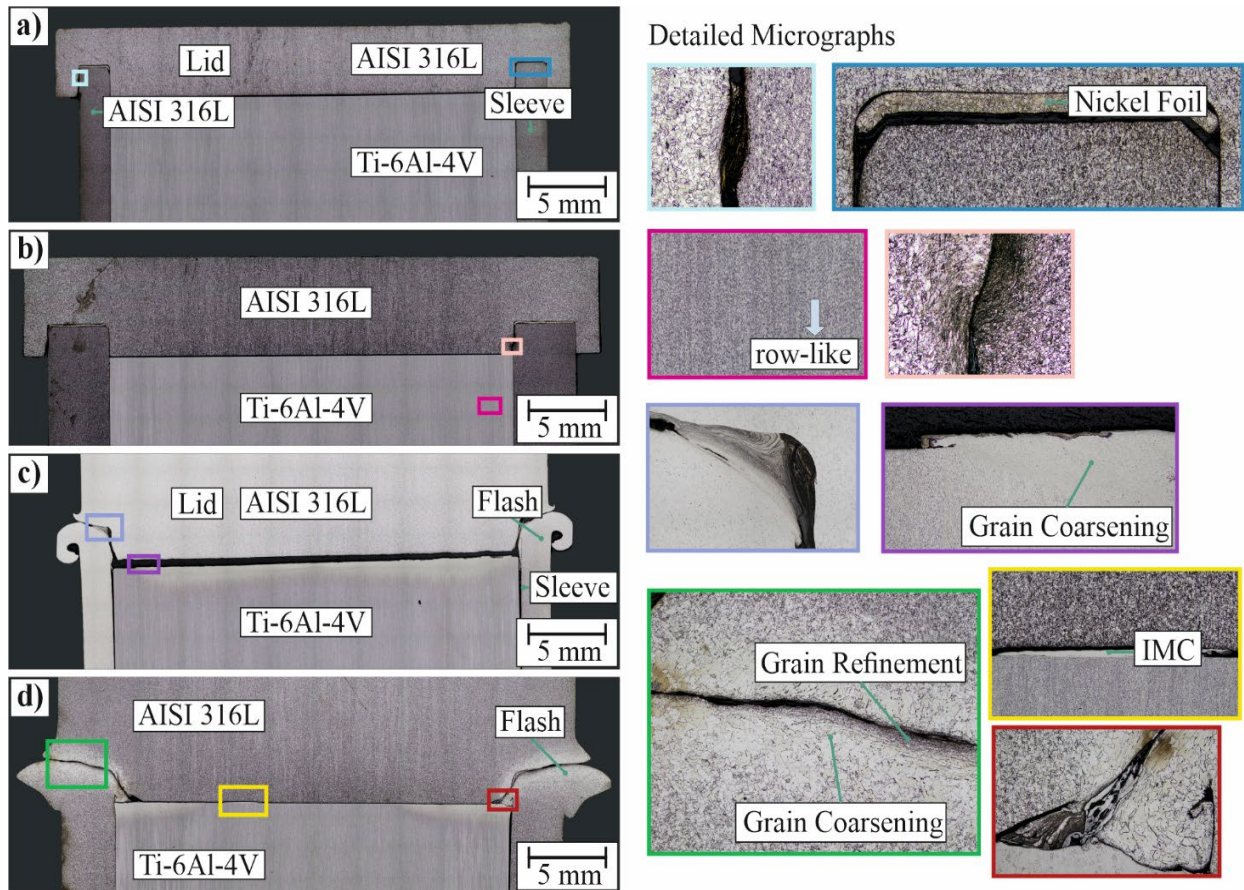
## Results and Discussion

### Investigation of encapsulated titanium billets.

Initially, the joining zones in the encapsulated titanium billets were examined before forging to ensure proper connection (see Fig. 3). The cross-sections of the diffusion-bonded capsules (see Fig. 3 a, b) show that the nickel foil did not improve the joining. This could be attributed to the insufficient contact between the joining partners. No bonding occurred in the horizontal section of the groove, which can be attributed to the existing interference fit. In the vertical areas between the lid and the sleeve, both capsules demonstrated partial joining. There is no visible IMC between the titanium billet and the capsule. Previous experiments observed this layer at temperatures above 700 °C, these parameters led to deformation of the capsule. The titanium core's microstructure indicates a row-like alignment in the vertical direction (see pink framed detail image) due to its manufacturing process. No grain coarsening occurred in the titanium core, unlike in the friction-welded samples exposed to increased heat input for a shorter time. Overall, the micrographs suggest that joining between the capsule shell and lid occurred, but not at the expected location in the upper groove area. In future experiments, the nickel foil could be introduced vertically to enhance bonding. The sleeve's thickness in this area should also be adjusted appropriately.

The RFW of the capsule with its corresponding lid successfully created a joint. The cross-sections of the RFW samples show variations in flash formation depending on the wall thickness of the steel capsule (see Fig. 3 c, d). With a capsule wall thickness of 2 mm, the desired swelling of the sleeve is evident, and the detailed view reveals the intermixing of the two joining partners. At the same friction force, a longer friction duration of an additional 5 s occurred with the thicker wall. This resulted in grain coarsening around the joining zone due to the extended heat input and hence local overheating. The grain refinement observed during RFW near the joining zone can be attributed to frictional heating and severe plastic deformation [15]. The titanium structure still displays its initial row-like grain alignment, resulting from its manufacturing process. However, in the upper titanium region near the joining zone, a change in the structure is visible, with grain coarsening present (exemplary seen in violet framed detail image). This is due to the temperature input during the RFW process. Additionally, an intermediate layer is sometimes visible, attributed to IMC, which appear to be localised (see yellow detail image). Due to the RFW, the capsule has bulged, and the interference fit has loosened. The stress from RFW likely caused slight deformation of the capsule. Notably, in the 4 mm capsule, a different gap width is observed when comparing both sides.

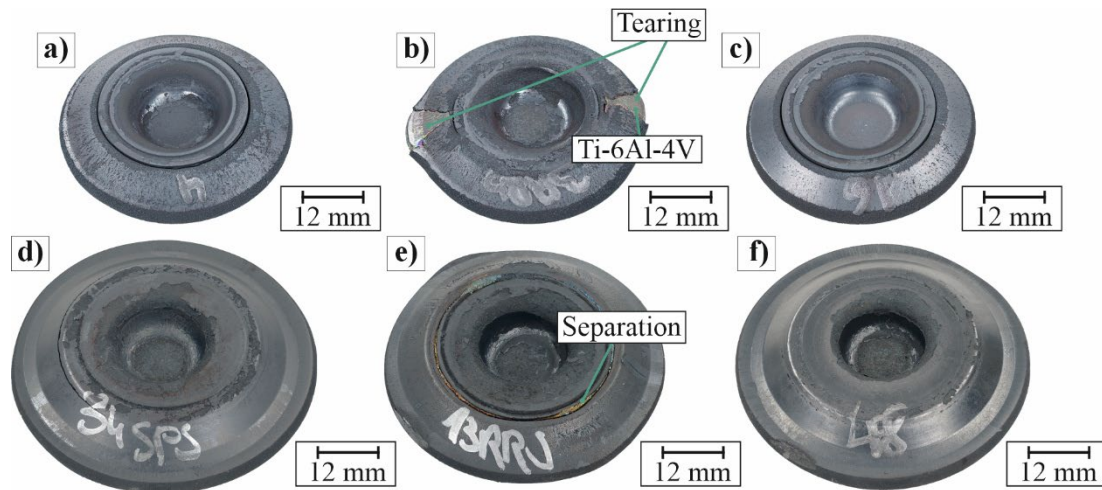
Both joining processes result in a bond between the capsule shell and the lid. The formation of an alpha-case could not be detected in the edge region. The bond of the RFW capsules appears more consistent compared to the DB capsules. Additionally, the process times are significantly shorter. Overall, both methods effectively achieve the sealing of the titanium billets.



**Fig. 3.** Overview micrographs of the joined capsules: (a, b) diffusion bonded and (c, d) friction welded, with wall thicknesses of 2 mm (a, c) and 4 mm (b, d). Detailed micrographs assigned to the positions in the overview image with coloured frames.

### Photographs of the forged components.

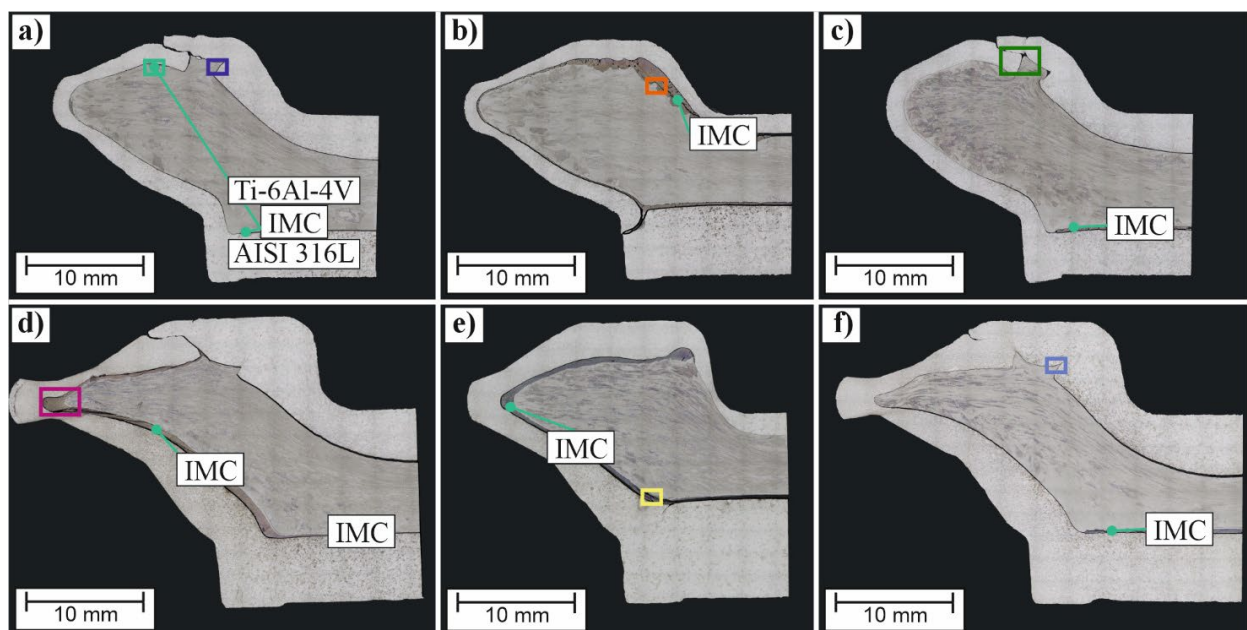
During the heating of some GP capsules, the lids separated from the sleeves. This was likely due to gases within the capsule expanding. Residual solvents from the graphite coating that did not fully evaporate could also be a cause. During heating in the furnace, these solvent vapours may have evaporated and expanded, pushing open the lid. To verify this, the graphitised titanium core could be exposed to elevated temperatures in the furnace to ensure complete removal of any remaining solvent residues. Overall, the capsules remain intact after forming (see Fig. 4), with the exception of the RFW capsule with a 2 mm wall thickness. The welded RFW capsules are designed with tapered edges to aid removal from the adapter. Damage in the form of tearing was observed in the thinner areas (b). As a result, the titanium in these areas is not protected against oxidation. However, with a wall thickness of 4 mm, no such cracks are present. An exemplary component of the joined capsules is shown, where the joining zone of the friction-welded sample was subjected to different stresses: one facing upwards during forging (e), experiencing more stress, and one with reduced stress placed with the joining zone at the bottom of the die (b). Sample (e) exhibits separation between the lid and sleeve all around the mandrel imprint. Titanium with distinctive discolouration appears, explained by tensile stresses from the mandrel indentation. The discolouration indicates the oxidation of the titanium. No additional cracks or external differences were observed in the capsules. To ensure complete encapsulation, the joining zone of friction-welded samples should be positioned in the less-stressed lower die. Due to the tapering for adapter removal, the 2 mm thick RFW capsule is unsuitable for forming titanium without atmospheric contact.



**Fig. 4.** Photographs of the forged components with a wall thickness of 2 mm (a-c) and 4 mm (d-f). The capsules are sealed differently: (a, d) DB, (b, e) RFW, and (c, f) GP.

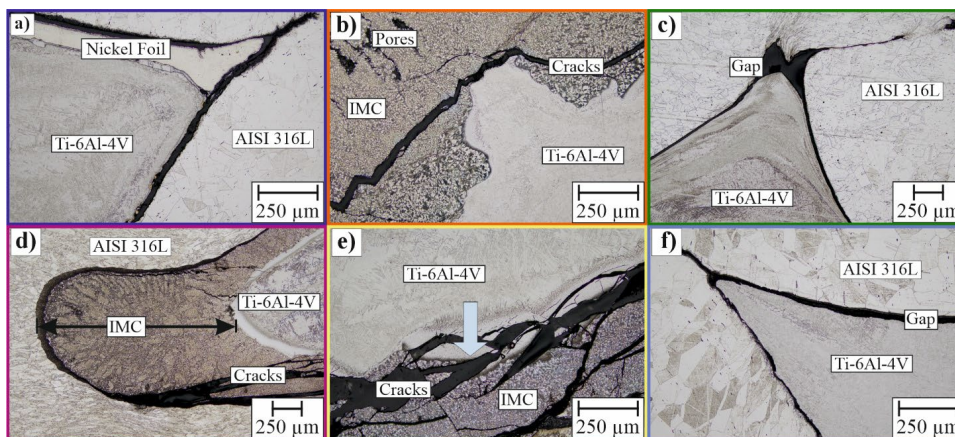
### Microscopic examination.

Using the cross-sections, the material flow of the encapsulated titanium components can be observed (see Fig. 5). Coloured squares in the figure indicate the positions of the detailed images (see Fig. 6). In the DB and GP capsules, the sleeve has detached from the groove, but the capsule remains sealed via the outer groove area, with titanium flowing into the gap. In the RFW samples, particularly 4RFW (Fig. 5e), the capsule has closed further due to pressure. However, a gap is visible in the joining zone of the 2RFW sample. It is important to note that this is a cross-section, and despite the rotational symmetry of the components, local differences in material flow and phases may exist. For instance, uncertainty in handling and position can affect flash formation and material distribution across the section. A further phase can be seen between the titanium and steel capsule, which literature identifies as IMC: TiFe, NiTi<sub>2</sub>, and complex oxides [16]. Overall, material flow within the same capsule thicknesses is comparable. The dies were designed in preliminary work to yield the same titanium core despite varying steel thicknesses. This is not achieved here, likely due to an inaccurate setting of the reversal point. The capsules protect the titanium core from the surrounding atmosphere during heating and forming, as they remain intact and there are no cracks.



**Fig. 5.** Overview micrographs of the cross-sections of the forged components with a wall thickness of 2 mm (a-c) and 4 mm (d-f). The capsules are sealed differently: (a, d) DB, (b, e) RFW, and (c, f) GP.

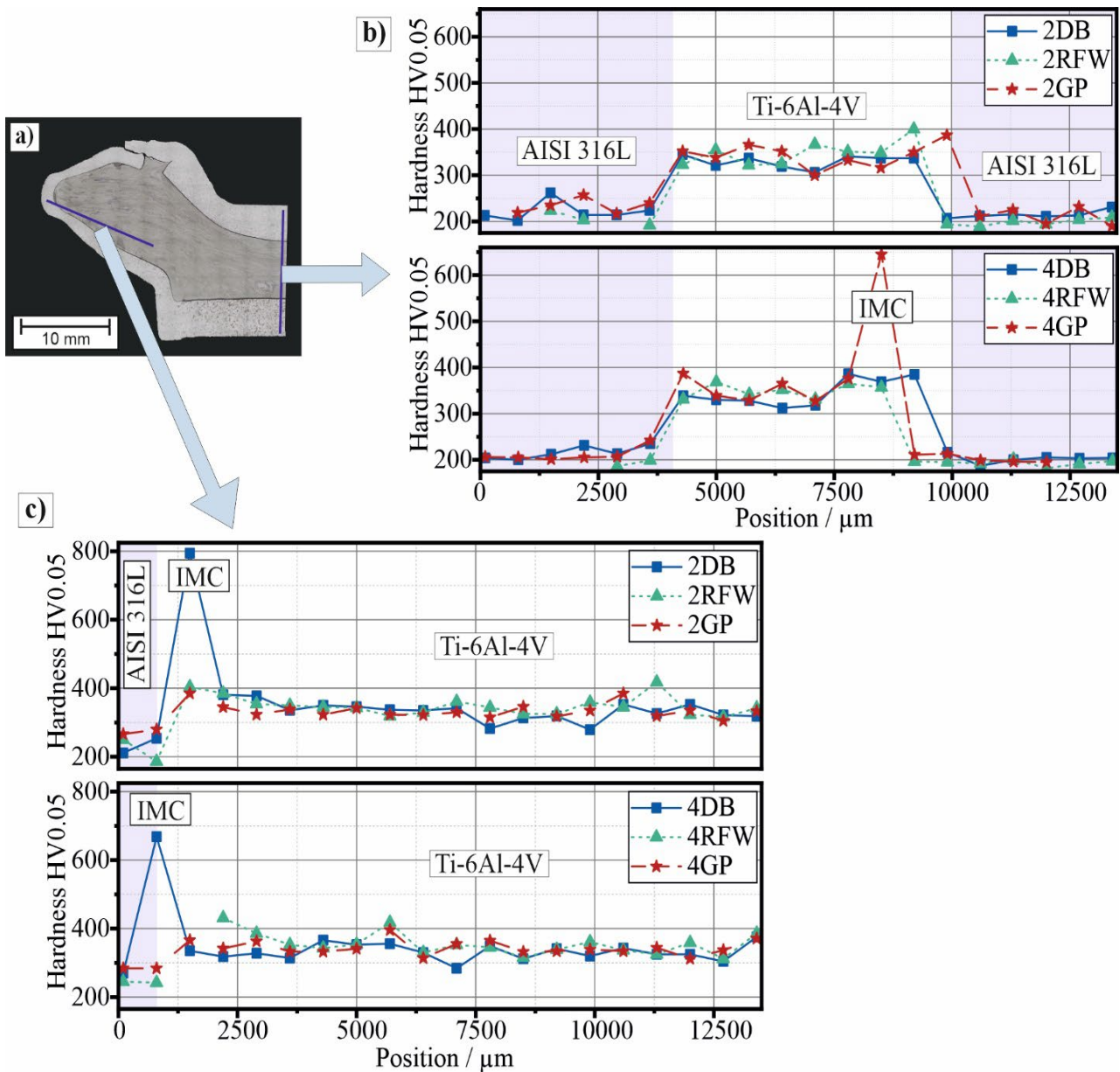
To analyse the forged components in detail, detailed images of the cross-sections are investigated, see Fig. 6. In specimen 2DB (a), the nickel foil is visible in the former groove area of the lid. After forming, the casing has separated from the lid's groove, with some titanium flowing into this area. A further intermediate layer is visible between the titanium and the nickel foil, resulting from their reaction. The titanium presents no alpha-case, indicating successful oxidation prevention. Specimen 2RFW (b) shows a distinct layer of intermetallic phases in the mandrel area, away from the friction welding zone. The thicker IMC compared to other samples may be due to the rotation of the titanium core during RFW, leading to early IMC formation. The IMC is riddled with cracks and pores (see Fig. 6 b), attributed to the phase's brittleness especially of TiFe and Fe-Cr-Ti [17]. The pores may result from diffusion processes linked to the Kirkendall effect [17]. This level of IMC formation is undesirable since it requires removal from the forged titanium, increasing process complexity and material waste. A potential solution in this variant could be the use of a separating agent to reduce IMC formation. Specimen 2GP (c) indicates IMC formation only in the lower area where the titanium core and capsule are in contact with each other. Graphite as a separating agent prevents further IMC formation. It is possible that the graphite application was insufficient in those areas. Overall, compared to specimen 2DB, there is a more even thinning of the steel capsule, likely due to reduced friction between the capsule and the titanium core through graphite lubrication. The detailed image reveals no alpha-case, indicating effective oxidation prevention. It also shows that high deformation rates can be achieved without the titanium cracking. Furthermore, the joining between the lid and housing is visible. The simple pressing of the titanium ensures isolation from the surrounding atmosphere with the chosen geometry. Specimen 4DB (d) exhibits a pronounced IMC along the entire interface between titanium and steel. This could be caused by IMC formation occurring during the diffusion process for capsule joining in this area. The IMC develops distinct regions and is interspersed with cracks. Consequently, upon capsule removal, separation between capsule and titanium core, as observed by Careau et al., is anticipated in the flash region [9]. Specimen 4RFW (e) also displays a pronounced IMC layer, which is more uniform compared to 2RFW. This may result from locally varied deformation during friction welding and the resulting different contact conditions. The detailed image shows the separation between titanium and IMC, as well as emerging gaps. In this area, a clear separation between IMC and titanium is visible, desirable for decapsulation to minimise IMC removal. An alpha-case is not observed in this specimen either. Specimen 4GP (f), like 2GP (c), exhibits reduced IMC formation. In the GP samples, an IMC formed exclusively between the capsule base and the titanium. This can be attributed to the prolonged contact during heating. During this period, the applied graphite spray does not effectively prevent the formation of the IMC. However, in all other areas that come into contact during forging, the graphite spray is sufficient to prevent the intermediate layer. The detailed image clearly illustrates the material flow without crack formation. Overall, encapsulation prevents alpha-case formation in all specimens, and IMC formation depends on contact conditions and can be utilised for later decapsulation.



**Fig. 6.** Detailed micrographs of the cross-sections of the forged components with a wall thickness of 2 mm (a-c) and 4 mm (d-f). The capsules are sealed differently: (a, d) DB, (b, e) RFW, and (c, f) GP. The coloured border indicates the corresponding position marked in Figure 5.

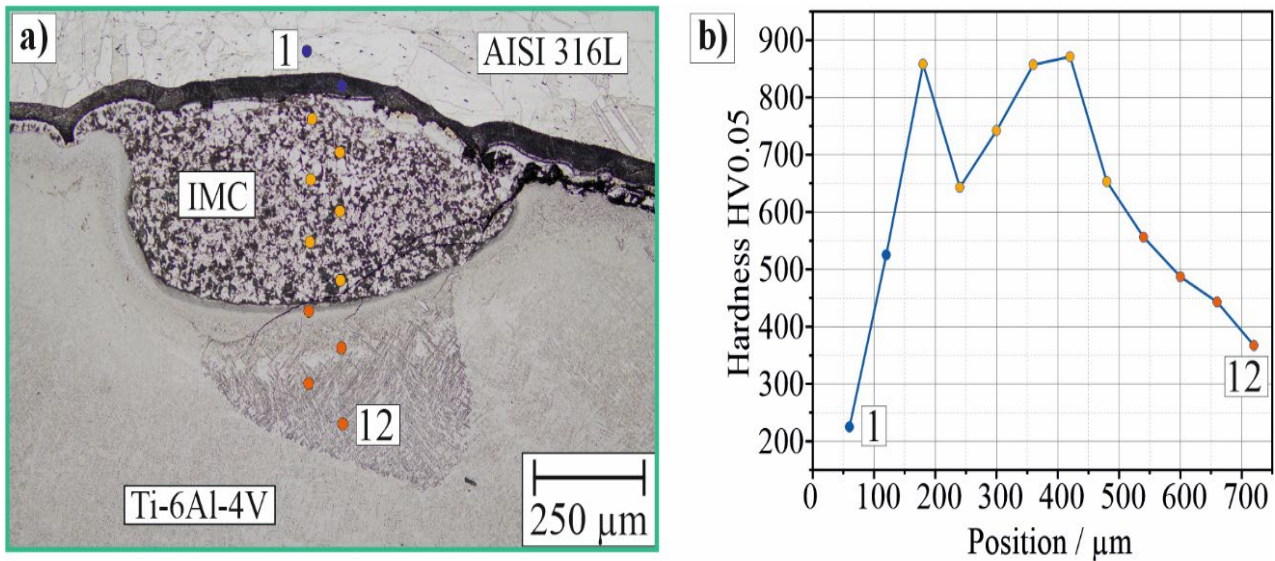
### Hardness Investigation.

To further investigate the mechanical properties of the forged component, hardness tests are conducted. These tests are performed at the positions marked in Figure 7a for the sample variations. As expected, the hardness profiles reveal that AISI 316L has a lower hardness compared to Ti-6Al-4V (see Fig. 7b, c). According to previous investigations, the hardness for Ti-6Al-4V is around 350 HV0.05 [10], as in this study. When measuring from the impact point of the mandrel to the bottom of the capsule, there is an increase in hardness in the base area (see Fig. 7b). This is attributed to the intermetallic phases present. Within the titanium core, the hardness fluctuates due to varying phases, true plastic strains and hence, dislocation densities. Also, when measuring from the flash area into the titanium core, the formation of IMC at the DB samples is visible externally. Overall, fluctuations are observed in the titanium core. No significant difference can be discerned between different wall thicknesses and thus different temperature distributions during forging, or their impact on hardness.



**Fig. 7.** a) Schematic illustration of hardness measurement positions, b) hardness measurements from mandrel area to capsule base, c) hardness measurements from flash area into the interior of the titanium.

To investigate the IMC, Fig. 8 illustrates an example of an IMC and the corresponding hardness profile. Generally, different layers can be observed. Starting from the steel, a darker layer is visible, which has increased hardness compared to the steel. The next layer features a structure of light and dark grains. These various phases likely have different hardness levels, which would explain the drop in hardness at position 4. As the titanium is approached, the hardness continues to decrease. Overall, the IMCs that have formed exhibit varying characteristics depending on the sample preparation. It is expected that the IMC will fracture at the point of highest hardness. According to this study, this will occur at the interface between titanium and the IMC, and consequently, after decapsulation, the IMC will already be separated from the titanium component. This is crucial for efficient decapsulation to minimise the necessary post-processing. This should be investigated in subsequent decapsulation trials.



**Fig. 8.** a) Detailed positioning of hardness measurements for sample 2DB, b) results of the hardness measurements.

### Summary and Outlook

In conclusion, the die forging of titanium billets encapsulated in steel was conducted, investigating an approach for isothermal forging conditions. The focus was placed on different encapsulation methods, the varying fit between titanium and steel sleeves, and different steel wall thicknesses to influence heat distribution within the titanium billet during forging. Encapsulating the titanium billets prevents pronounced oxide layers and the formation of an alpha-case, as seen in non-encapsulated forging. The selected lid geometry in these experiments provides protection against ambient gases. A 2 mm thick steel wall encapsulation is sufficient to isolate the titanium from ambient gases. As no beneficial effects on material flow and microstructure are observed with thicker encapsulation, it is advisable to avoid the thicker encapsulation for material and energy efficiency. Depending on the contact conditions, different thicknesses of IMCs are formed, which can with the exception of the capsule base be prevented by introducing graphite as a separating agent, resulting in a somewhat more uniform material flow.

The different formation of IMC allows for a comparison of the decapsulation process with and without IMC in future studies. Further investigations will examine alpha-beta forging, where temperature distribution is even more critical. Thus, more significant differences are expected with different capsule wall thicknesses. Additionally, other separating agents like glass coating or alumina particles can be used to prevent IMC formation in the lower capsule area.

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