# Comparison of HIP Composite and HIP Solid Material with Melting Metallurgically Produced Solid Material

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**Abstract.** The production of composite materials using Hot Isostatic Pressing (HIP) technology is finding more and more applications. Composite parts for co-rotating twin screw extruders have been introduced in practice for years and are indispensable. Further applications in the roller sector for the processing of high-quality sheet metal and wood pellet production contribute to quality improvement and service life extension and can be tailored precisely to the demands. Composite material is particularly advantageous for large dimensions. Requirements such as high hardness in the rim zone and high strength in the load-bearing core can be combined with different materials. In the present work, HIP composite materials and HIP solid materials were compared with melting metallurgically produced solid materials. The differences in hardness and strength in the rim zone and in the core after heat treatment are presented, showing the advantages and disadvantages of both systems. The choice of composite material depends on the requirements and can cover contrary needs such as corrosion, wear resistance and high strength.

## Introduction

Hot Isostatic Pressing (HIP) was developed 1955 at the *Battelle Memorial Institute's Columbus Laboratories* in Columbus (Ohio, USA) initially for cladding nuclear fuel elements for submarines [1,2]. Since those days, HIP has established itself as a reliable manufacturing process for complex and highly stressed components in different industrial sectors, e.g., for the aerospace, offshore, mechanical engineering or energy sector [3,4].

As specialty, the diffusion processes taking place during HIP are used to produce composite parts and thus create tailored properties at those points of a tool where they are needed. In twin screw extruders for plastic processing, for example, the screws are made by a combination of a conventional tempering or tool steel with a high toughness in the core and a corrosion and/or abrasion-resistant powder metallurgical (PM) material in the rim zone. As a result, the tough core allows operation at high torques and thus ensures an increase in productivity while the PM layer enables service life to be extended by up to a factor of 10, depending on the chosen material and the materials to be processed [5].

In the production of metal sheets, highest requirements are placed on the surface integrity of the working rolls, since irregularities on the roll surface are transferred directly to the sheet, where they lead to intolerable deviations in shape and dimensions. To maintain surface integrity as long as possible, the use of suitable materials is essential. PM high-speed steels (HSS) are ideal for this purpose, as they have a special microstructural design leading to high wear resistance and, depending on their chemical composition, high corrosion resistance, and can therefore meet the demands in cold rolling mills to the highest degree. In this case as well, a composite with a tough core is used to absorb the alternating loads during the rolling process and to assure the longest possible service life of the rolls. Moreover, productivity can be improved by applying higher torques.

In order to withstand the mineral and fiber content of the wood, acting like lubricating paper to pellet rollers and pan grinders, a HIP composite solution can be used. Here, also, a combination of two different requirements have to be met. Hard on the outside, solid and tough on the inside. This ensures to improve wear resistance while absorbing the high pressing forces. Considerable service life improvements by a factor of 3 and more have been achieved [5].

Now to demonstrate and compare the special properties of a HIP composite to solid material parts, in this study three bars, namely a solid bar made of PM HSS, a composite bar of PM HSS/tool steel and a solid bar of conventional HSS are produced and hardened. The advantages and disadvantages of the different systems are shown by comparing the transverse rupture strength (TRS) and hardness profiles of the components.

## Materials and Experimental Setup

The parts studied in this contribution consist of the following materials:

1) Solid bar made of powder metallurgically produced HSS *PM 1.3344* (SARAMET 23, AISI M 3-2 PM)
2) Composite bar made of *PM 1.3344* in the rim zone and melting metallurgically (MM) produced tool steel *1.2344* (AISI H13) in the core.
3) Solid bar made of melting metallurgically produced HSS *MM 1.3344* (AISI M 3-2).

The chemical compositions of the used materials are presented in Table 1. The differences between PM 1.3344 and MM 1.3344 lie in the microstructure, particularly in the grain size as well as in the size and distribution of the carbides, with PM 1.3344 having a significantly finer-grained microstructure in which numerous small and homogeneously distributed carbides are embedded in the matrix, c.f. Fig. 1 (a). In contrast, the conventional production route via ingot casting causes considerably bigger grains as well as coarse and linearly arranged carbides in MM 1.3344, which is illustrated in Fig. 1 (b). This microstructural texture can be further intensified by the following machining steps, while the HIP process ensures an isotropic microstructure in PM 1.3344 [6].

The bars were produced in the dimensions Ø 200 x 500 mm, c.f. Fig. 2. In the case of the composite bar, the capsule was first fitted with a solid bar of 1.2344 with a diameter of 140 mm, and then filled with PM 1.3344 powder. After mechanical pre-densification, sealing and evacuation, the PM 1.3344 and the composite bar capsules were hipped together in one load with the parameters 1100 bar/1150 °C/4 h/cooling 40 °C/h. After checking the argon content [7] and release of the manufactured bars, the capsule material was mechanically removed via turning. The MM 1.3344 bar and the 1.2344 core bar were purchased commercially in the rolled and machined condition.

Subsequent to the HIP process a heat treatment under vacuum atmosphere was conducted. All three bars were subjected to the following heat treatment in one furnace load:

- Hardening: 1130 °C/45 min/pressurized nitrogen cooling down to 350 °C/100 min holding to enable temperature balance between rim zone and core/pressurized nitrogen cooling to RT

*Table 1: Chemical compositions of the used materials listing the main alloying elements in wt.%*

| Material | C [%] | Si [%] | Cr [%] | Mo [%] | W [%] | V [%] | Fe [%] |
|---|---|---|---|---|---|---|---|
| PM 1.3344 | 1.3 | 0.5 | 4.2 | 5.0 | 6.4 | 3.1 | Bal. |
| 1.2344 | 0.4 | 1.0 | 5.3 | 1.4 | - | 1.0 | Bal. |
| MM 1.3344 | 1.3 | 0.3 | 4.0 | 5.0 | 6.2 | 3.0 | Bal. |

- Tempering: 530 °C/4 h/air cooling
500 °C/4 h/air cooling
500 °C/4 h/air cooling

After heat treatment a plate with a height of 35.2 mm was cut out of the center of each bar via wire-EDM, shown exemplarily for the composite bar in Fig. 2. During EDM, the MM 1.3344 bar cracked, see Fig. 3, which is a result of massive residual stresses introduced into the bar by the hardening process. However, the crack doesn't influence the mechanical testing of this bar. In contrast, the PM 1.3344 bar and the composite bar could be machined without any problems. Due to the fine-grained, segregation-free microstructure of the PM material, the microstructural transformation and precipitation processes during heat treatment occur much more homogeneously than in the MM material, resulting in significantly lower hardening stresses.

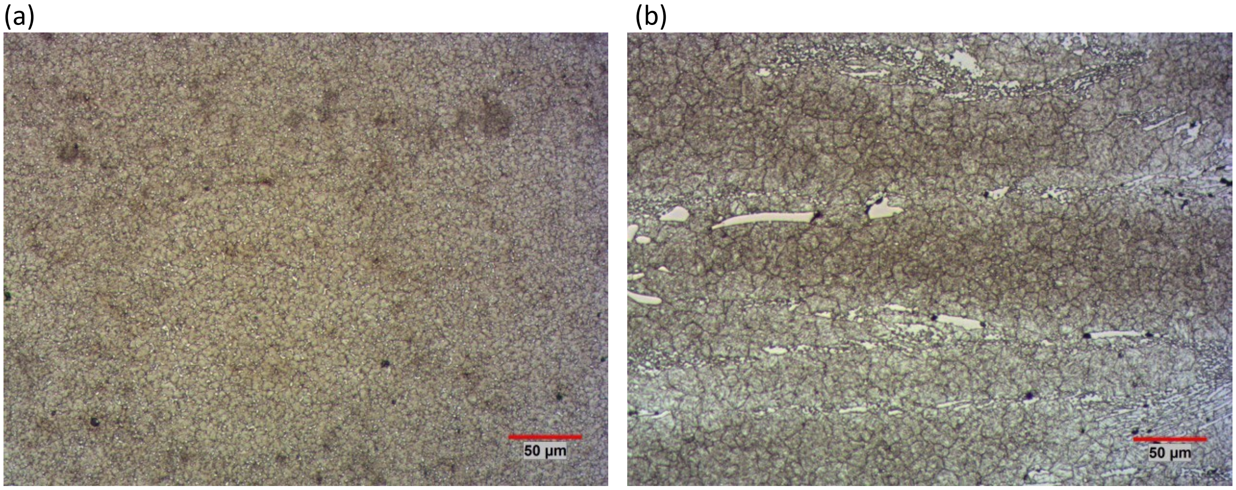

*Figure 1. Micrographs showing a fine-grained microstructure with numerous small, homogeneously distributed carbides in PM 1.3344 (a) and bigger grains with coarse, linearly arranged carbides in MM 1.3344 (b)*

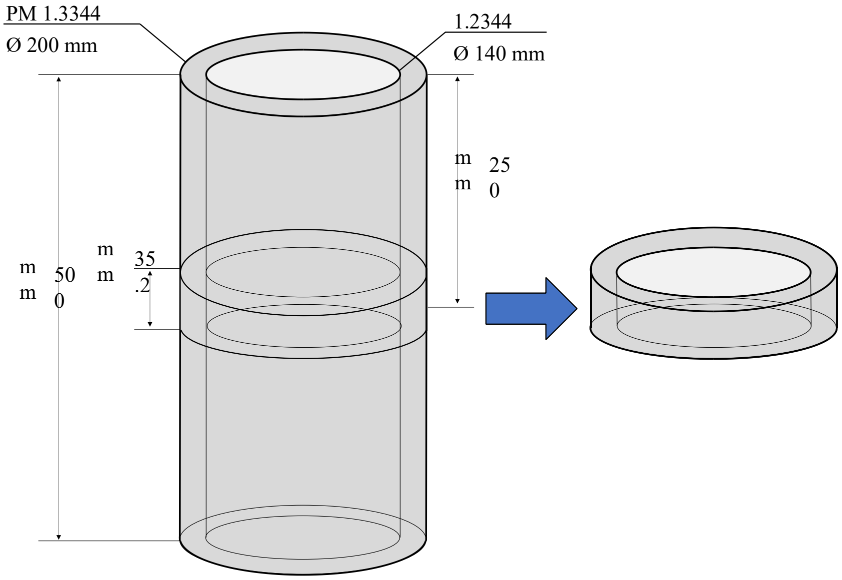

*Figure 2. Bar dimensions and plate extraction out of the center, exemplarily shown for the composite bar.*

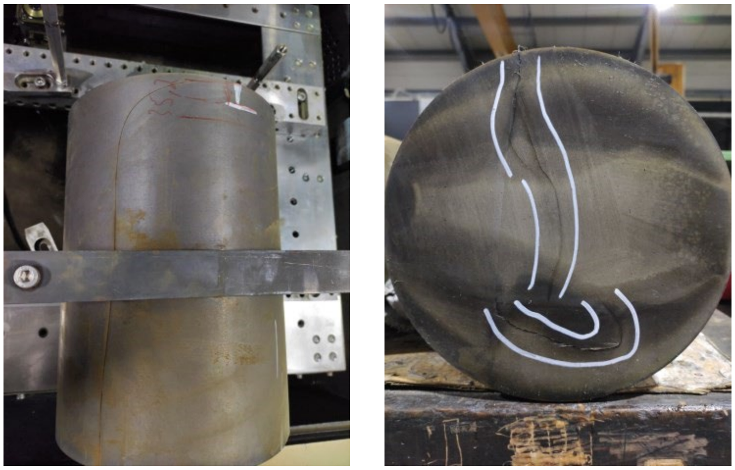

*Figure 3. The stresses introduced by the heat treatment cause the MM 1.3344 bar to crack during EDM.*

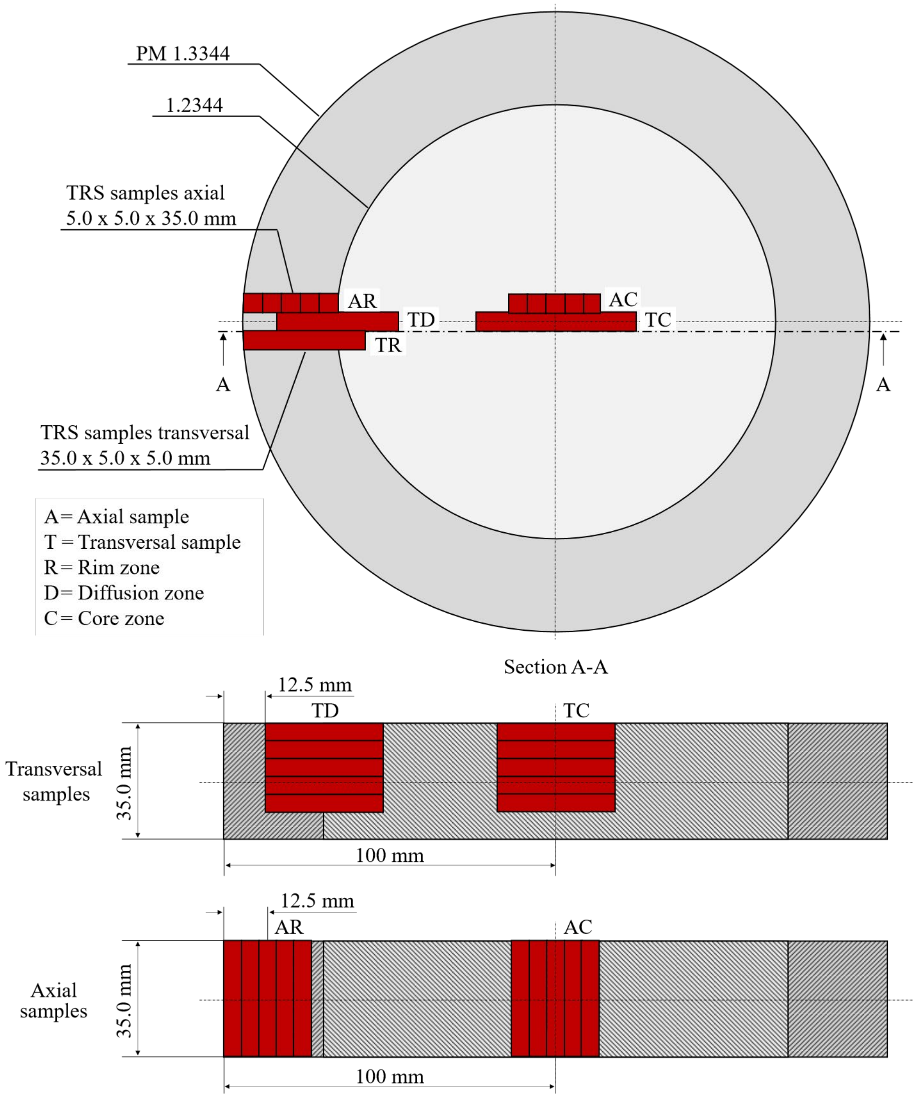

*Figure 4. Sampling plan illustrated by the example of the composite bar PM 1.3344/1.2344*

In order to measure the hardness profile, both in x- and y-direction six hardness measurements were performed close to the edge and every 10 mm over the cross-section of the components using an *Equotip 550* mobile hardness tester.

To check the strength characteristics of the three bars, TRS tests were performed acc. to DIN EN ISO 3327 [8]. Axial (A) and transversal (T) samples were eroded out of different depths, from the rim zone (R) over the diffusion zone (D) to the core (C), and ground on a surface grinding machine. The sampling plan in Fig. 4 shows that 25 samples were manufactured out of each plate.

## Results and Discussion

The hardness measurement results over the cross-section are presented in Fig. 5. The mean values at each measuring depth are depicted in the form of columns. The measurement scatter is recorded on basis of the standard deviation indicated by error bars.

The PM 1.3344 solid bar, Fig. 5 (a), exhibits a homogeneous hardness profile up to the component core. Over all measuring depths the hardness values vary between 61.6 and 63.6 HRC. This small scattering is an indication of a homogeneous hardening microstructure, which results in uniform mechanical properties over the entire cross-section of the bar.

The results in Fig. 5 (b) show that the composite bar reveals a hardness of 62.1 - 63.1 HRC in the PM 1.3344 layer up to a measuring depth of 30 mm, which is on the same level as the PM

1.3344 solid bar. All measurements above 30 mm are made within the range of the conventional tool steel 1.2344. Here the hardness values experience a significant drop to 57.0 HRC and fall down to 55.3 HRC with increasing distance from the surface.

In the case of the MM 1.3344 solid bar, presented in Fig. 5 (c), the hardness values are at a comparable level to PM 1.3344. However, it can be seen that the hardness profile scatters more widely with values between 59.5 - 63.1 HRC. Additionally, the measurements within the individual measuring depths also scatter more widely, caused by microstructural inhomogeneities coming from the melting metallurgical manufacturing process.

The TRS test results are illustrated in Fig. 6. Each column represents the mean value of five measurements on every sample. The standard deviation is featured again by error bars. In the case of the PM 1.3344 solid bar, represented by the blue columns, the TRS shows no dependence on the location or the orientation of the sample. The hot isostatic compaction of the metal powder ensures a fine-grained, homogeneous microstructure, which allows uniform hardening right into the component core on the one hand, and isotropic material properties on the other hand, so that the values for transversal and axial samples are at the same level. In addition to high absolute values, this results in an extremely low scattering of the hardness values of less than 3 %.

The results of the composite bar are depicted in orange columns in Fig. 6. In the zone TD, the samples are positioned in such way that the test load is applied at the boundary between PM 1.3344 and 1.2344 (diffusion zone). All samples of this section did not break in the diffusion zone but in the region of the 1.2344 bulk material. Thus, at TD, the column characterizes the TRS of the high toughness 1.2344 tool steel, which achieves comparable values to PM 1.3344. The samples at positions TR and AR are located almost completely within the range of PM 1.3344 and show therefore similar measurement results to the PM 1.3344 solid bar. The samples from positions TC and AC lie entirely in the range of 1.2344. The measurement results at position TC are about 8 % below the values of TD. This could indicate that the grains get coarser from the rim to the core because of different deformation ratios during the rolling process, which is explained more in detail for the MM 1.3344 bar in the following section. The axial samples at this depth AC provide about 3% more in TRS compared to the transversal samples TC. This can be explained by the microstructural texture resulting from the manufacturing process of the bar. During rolling, the grains get stretched in rolling direction which leads to the formation of the so-called "forging-fiber" that runs through the core bar in axial direction. As a result, during the TRS test, the axial samples are loaded perpendicular to the forging fiber and can therefore offer greater resistance to breaking than the transversal samples which are loaded parallel to the forging fiber. This influence is reduced by coarser grains in the core area, resulting in this slight difference of only 3%.

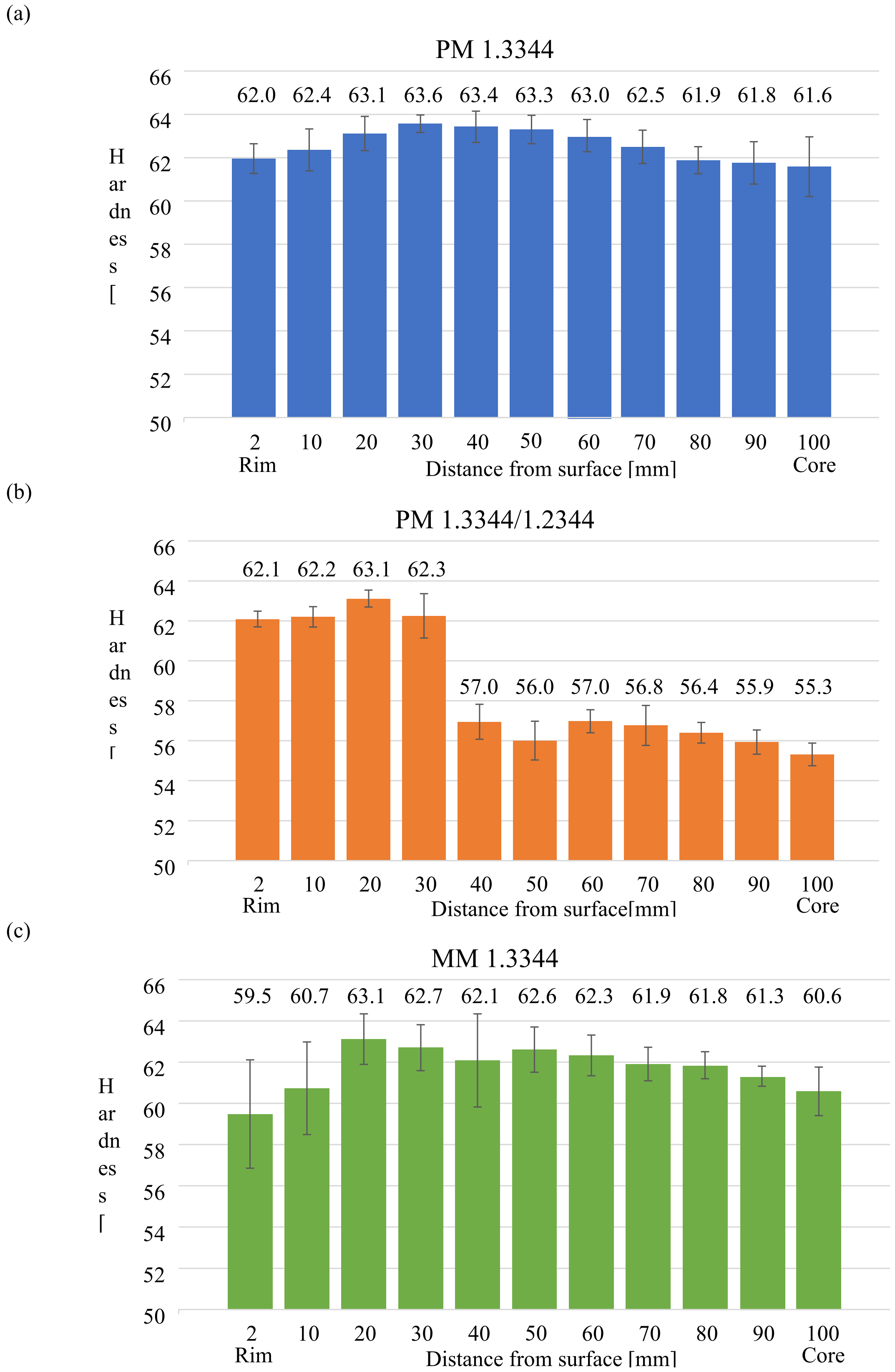

*Figure 5. Hardness profile over the cross-section for PM 1.3344 solid bar (a), PM 1.3344/1.2344 composite bar (b) and MM 1.3344 solid bar (c). At each measuring depth 12 measurements were performed using a mobile hardness tester*

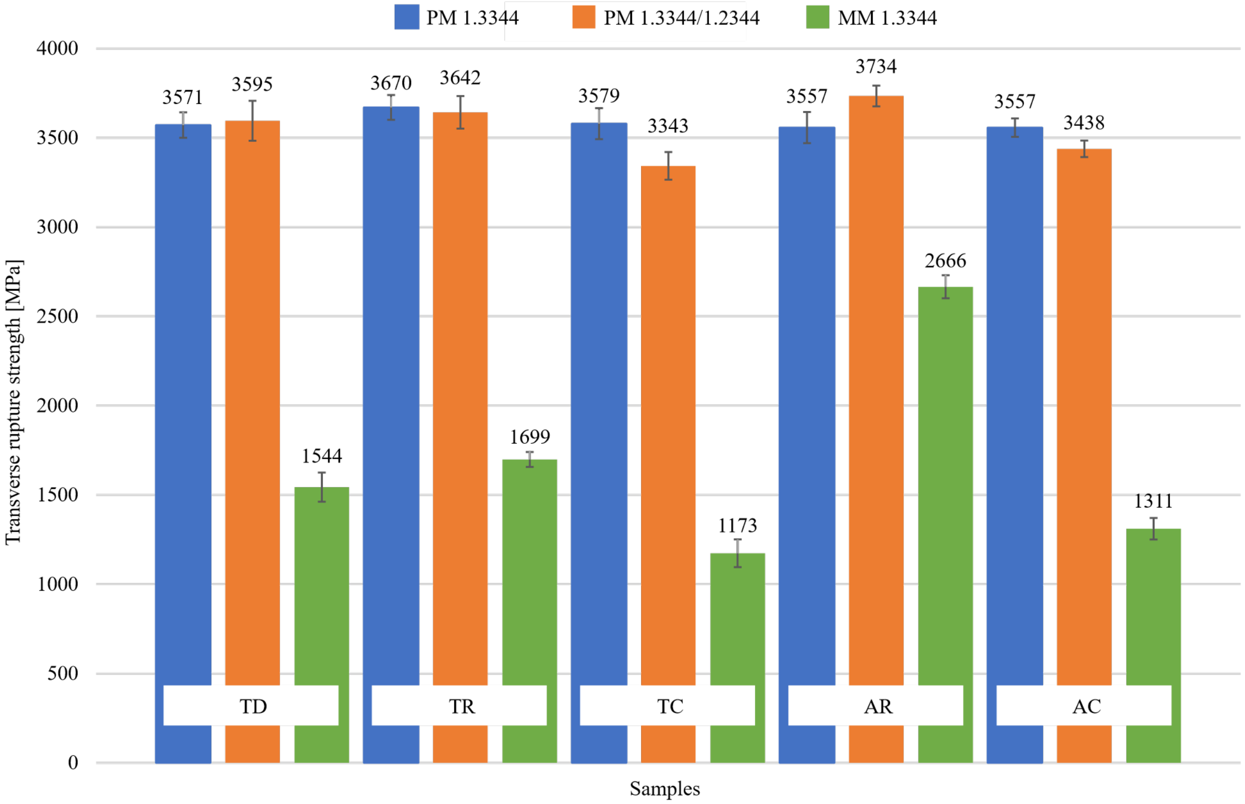

*Figure 6. TRS measurement results*

The measurement results of the MM 1.3344 bar, green columns in Fig 6, show a significant dependence on the depth and the sample orientation. In the rim zone at position TR, 10 % higher values are measured compared to position TD and even 45 % higher values compared to TC in the bar core. In the case of the axial samples, this influence is even more evident. Here, double the values are measured in the rim zone AR compared to the core AC. As mentioned in the section above, this fact can be explained by differences in grain size between rim and core, c.f. Fig. 7. During the production of the bar, the rolling process causes a high deformation level in the area close to the rim. This deformation decreases further and further on the way to the core. The high deformation level ensures a high dislocation density in the rim zone. During the subsequent heat treatment, a high dislocation density initially leads to a high number of sub-grain boundaries (recovery) which in turn results in a high nucleation number for new grains (recrystallization) and finally in a fine-grained austenite structure. As a result, the fine-grained austenite enables a fine-grained quenched and tempered structure after completion of the heat treatment and thus a higher strength according to the Hall-Patch correlation.

In addition, examining the influence of the sample orientation, it can be seen that the axial samples AR of MM 1.3344 yield almost 57 % more TRS than the transversal samples TR at the same depth. This difference can be attributed to the orientation of the forging fiber in axial direction of the bar, as previously described. In the core area, the coarser grains weaken the orientation influence. Here, the axial samples AC lie only about 12 % above the transversal samples TC.

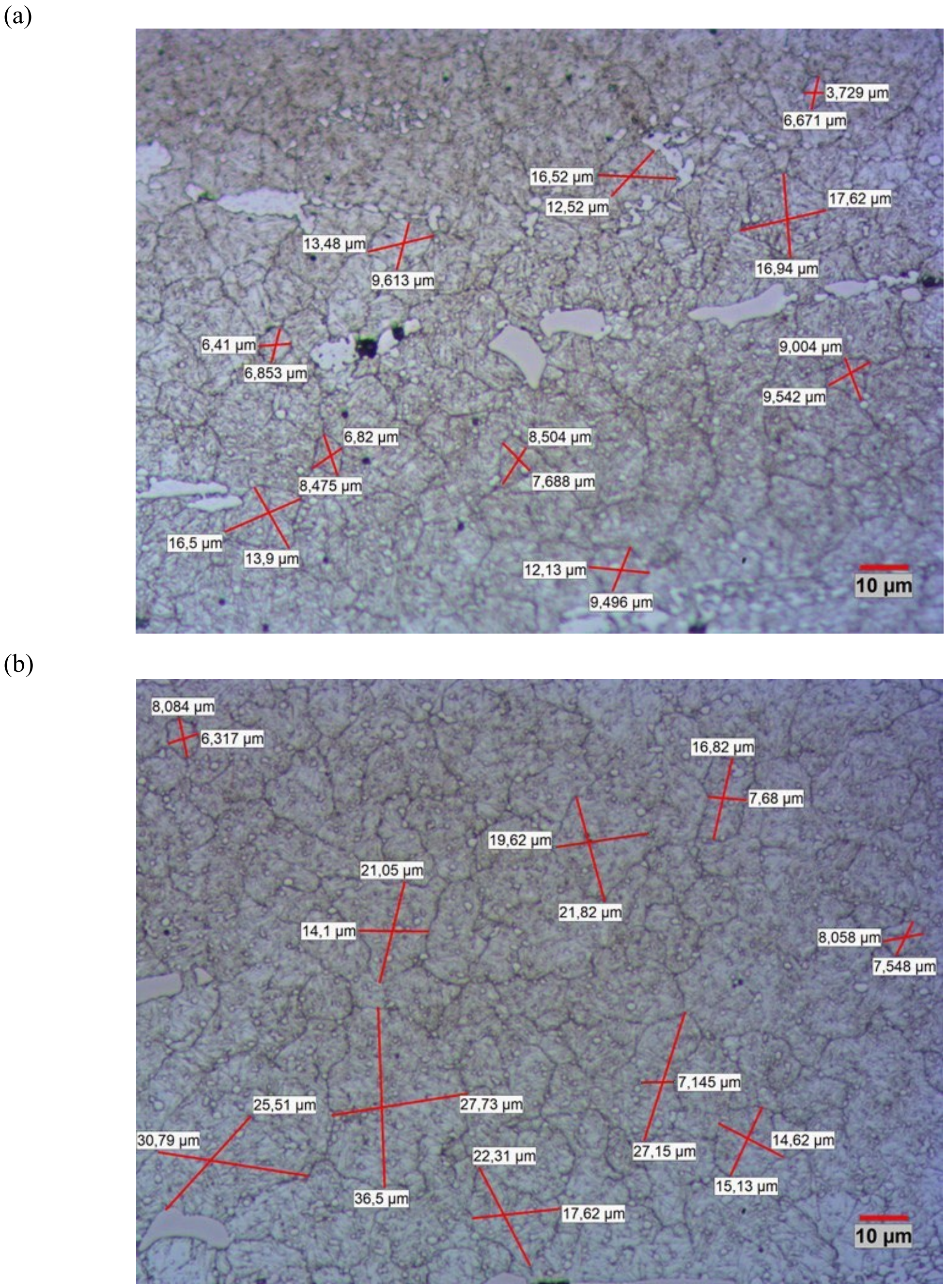

*Figure 7: Smaller grains in the rim zone (a) and coarser grains in the core (b) of the MM 1.3344 solid bar leading to higher TRS values in the rim zone and lower values in the core.*

Comparing the different materials, it is obvious that the PM 1.3344 exhibits considerably higher TRS values than its melting metallurgical counterpart MM 1.3344 across all measuring positions and orientations. In some cases, the values are even by a factor of 2 - 3 higher. Only at position AR the forging fiber reduces the difference between the two materials, although the PM 1.3344 values here still achieve 33% more in TRS. Compared with the highly ductile 1.2344 tool steel, PM 1.3344 shows comparable TRS values. This points out, that PM 1.3344 combines high toughness with high strength and hardness together.

## Conclusions and Outlook

In the present work, a PM 1.3344 solid bar, a PM 1.3344/1.2344 composite bar and a melting metallurgically produced 1.3344 solid bar were compared regarding the hardness and transverse rupture strength after heat treatment. The experiments point out that the PM 1.3344 has an exceptionally favorable combination of hardness, strength and toughness, coming from its fine-grained microstructure with a high amount of small and homogeneous distributed wear-resistant carbides, which underlines its suitability as working rolls in cold rolling mills or as wear parts in twin screw extruders, respectively pellet mills. This microstructure permits uniform quenching and tempering over the entire powder layer, which, in addition to a high hardness, results in high transverse rupture strengths, partly higher by a factor of 2 - 3 compared to its melting metallurgical counterpart MM 1.3344. Thereby the PM 1.3344 results depend neither on the orientation nor on the depth of the samples.

The composite PM 1.334/1.2344 and the solid bar PM 1.3344 hardly show any differences in their TRS values. This indicates that the heat treatment for the composite bar should be optimized to further improve the TRS of the core zone. In a first step the tempering temperature could be increased. This is expected to decrease the hardness and to increase the transverse rupture strength of 1.2344. Moreover torsion tests should be performed to better meet the alternating loads in the specific application. It is expected that the composite bar can withstand significantly higher torsional forces than the PM solid bar, resulting in considerable service life extensions. This fact has been proven in twin screw extruders for years. Regardless of the results of further investigations, at today's raw material prices a composite part made of expensive PM powders and cheaper conventional steels offers cost savings without compromising on quality.

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