Advanced Materialography

part of VERDER scientific

**ATM GmbH**
Emil-Reinert-Str. 2
57636 Mammelzen, Germany

Phone: +49 (0) 2681 9539-0
Fax: +49 (0) 2681 9539-27

E-Mail: info@atm-m.com
Internet www.atm-m.com

# Materialographic Preparation of Specimens Produced by 3D-Printing Technologies

## Introduction

Having been invented in 1983 and patented in 1986 by C. Hull the 3D printing technology (initially named "stereo lithography") is a well-established production method in today's industrial market. Forecasts predict an increase of 774 % in the global turnover during the next 5 years – which makes 3D printing one of the most prospering markets in the near future.

One of the various 3D printing methods is **additive laser powder build-up welding**. This technique is characterized by coating materials in powder form with the help of laser welding. The desired shape of the specific product is formed by following trajectories which are predefined prior to manufacturing. The energy of the laser melts the used metal powder forming a welding bead. The final geometry is given its three-dimensional contour by the overlapping of the welding beads based on the paths of the predefined trajectories. Optimization of the additive laser powder build-up welding focuses on economical processing with high quality and accuracy. Another focus lies on scalability: large scale on the one hand and implementing microstructures less than 100 µm on the other.[1]

The materials used for additive laser powder build-up welding are mainly:

- Light metal
- Nickel super alloys
- Steel
- Intermetallic materials
- Hard materials (carbides)

1 Fraunhofer IWS, Additive Manufacturing, 2016, www.isam.network

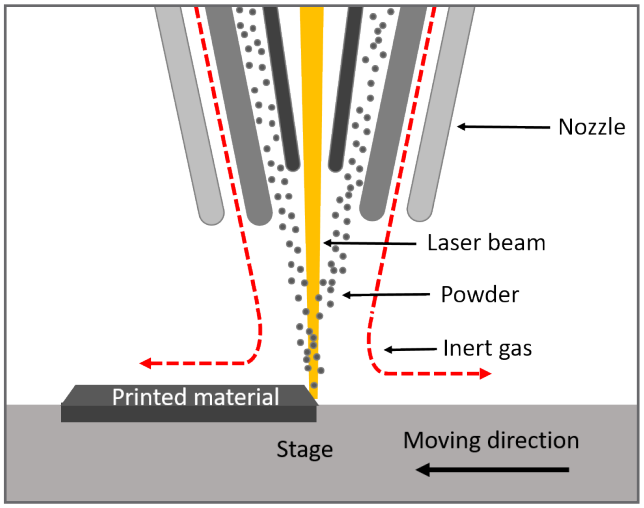


Figure 1: process of additive laser powder build-up welding.

## Materialographic Preparation Process

In the following, we will demonstrate the materialographic preparation process of a sample produced by additive manufacturing. In materialography, a sample taken from a work piece is called specimen.

**A typical materialographic examination includes the following steps:**

- Sectioning e. g. with an abrasive cutter
- Mounting which offers several advantages for further preparation
- Grinding/polishing for the preparation of the microstructure
- Examination by
- Image analysis
- Hardness testing

For this article a steel sample (X6Cr17, material number: 1.4016) manufactured by additive laser powder build-up welding was investigated. The first step was to obtain a smaller sample piece (=specimen) which is representative of the complete workpiece. This was achieved by using **ATM's Brillant 220 precision cutter** with a thin CBN (cubic boron nitride) blade (wheel thickness: 0.65 mm, wheel diameter: 153 mm) as shown in Figure 2.

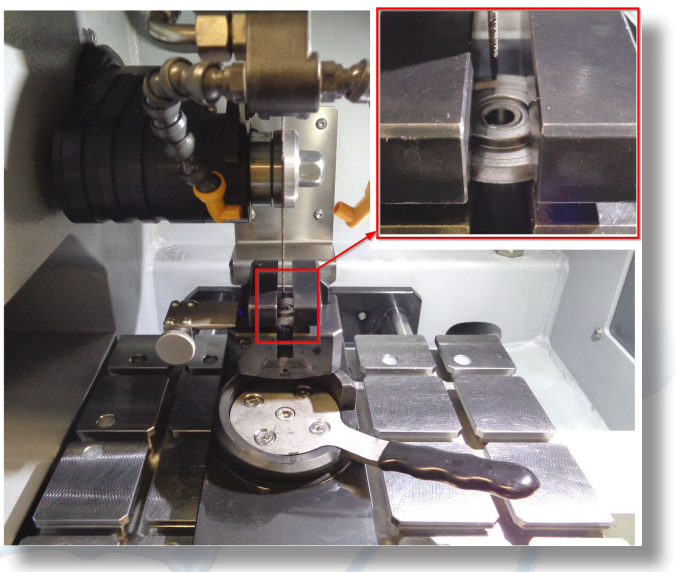

Figure 2: Brillant 220 machine setup. Detail: clamped sample (clamping tool: vertical vice single).

The cutting was effected with a pulsed direct cut (0.2 mm forwards and 0.2 mm backwards) with a feed speed of 1 mm/s and a rotational speed of 4500 rpm.

After cutting, the specimen was mounted in a hot mounting material (Epo black) with an **ATM Opal 460 hot mounting press** to obtain a specimen which is easier to handle. Mounting was carried out at a pressure of 200 bar for 6 minutes at 180°C, followed by a cooling cycle of 6 minutes. Another advantage is the high degree of parallelism of the mounted specimens of 51 µm ± 1 µm (the tolerances are based on the caliper used for height measurements of the specimens). The mounted specimens were ground (individual force) and polished (individual force) afterwards with a **semi-automated grinding and polishing machine, ATM's Saphir 550**. The grinding process was divided into two steps. The first one was plane grinding using a silicon carbide (SiC) grinding paper with grit size P240 to remove all deformations caused by the cutting process. This was followed by grinding with a SiC paper with grit size P600 to smoothen the surface for subsequent polishing steps. First, the specimen was pre-polished with the hard **Galaxy BETA polishing cloth** and 9 µm polycrystalline diamond suspension, followed by a **medium-hard cloth made of silk** (ATM: GAMMA) and 3 µm poly diamond suspension. The last step, called final polishing, was done on a **soft synthetic polishing cloth** (ATM: OMEGA) and Eposil M. The detailed preparation parameters are indicated in Table 1.

| Step | Medium | Lubricant/ suspension | Speed platen [rpm] | direction sample holder | Single load [N] | Time [min] |
|---|---|---|---|---|---|---|
| Grinding | SiC, P240 | Water | 150 | Clockwise | 30 | 1:00 |
| Grinding | SiC P600 | Water | 150 | Clockwise | 30 | 1:00 |
| Polishing | BETA | Alcohol, diamond 9 µm (poly) | 150 | Counter-clockwise | 35 | 4:30 |
| Polishing | GAMMA | Alcohol, diamond 3 µm (poly) | 150 | Counter-clockwise | 35 | 4:00 |
| Polishing | OMEGA | Water, Eposil M | 100 | Clockwise | 30 | 1:30 |

Table 1: Grinding and polishing parameters.

Based on this preparation sequence, a finely polished specimen surface was obtained. Figure 3 shows an image taken with an incident optical microscope (incident light) at a magnification of 100.

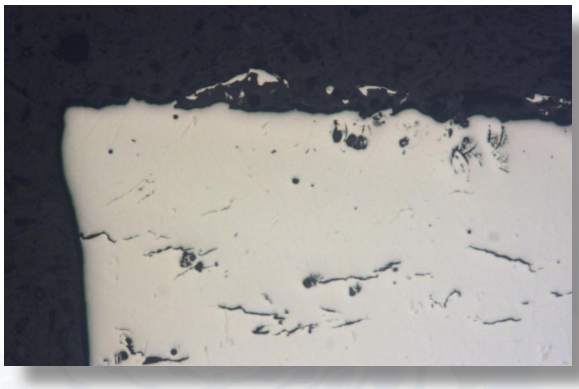

Figure 3: Image of the prepared specimen surface. Due to the polished surface the light is reflected almost equally and the microstructure is not discernible.

As the light is reflected almost equally over the whole specimen surface, the microstructure remains invisible. Due to the nature of the human eye, a minimum difference in contrast of 10% is needed to make the contrast visible on any surface. This contrasting is achieved by etching. In our example, the etchant “V2A Beize” for pickling was used to contrast the surface by selective etching of the different phases of the investigated X6Cr17 steel. Etching was done for 45 s and the microstructure is very well discernible as can be seen in Figure 4.

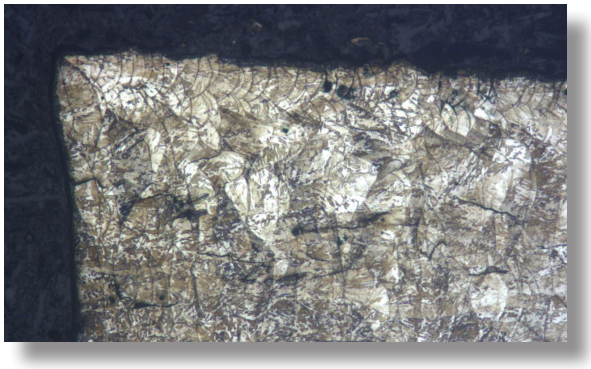

Figure 4: Etched specimen using "V2A Beize" (for 45 s). Edge section. The microstructure is clearly discernible.

The microstructure was also contrasted well in the middle of the specimen surface indicating that the whole prepared surface was successfully contrasted as shown in Figure 5.


Figure 5: Contrasted specimen. The welded-based microstructure of the manufactured workpiece is clearly visible.

Further examinations, like **hardness testing**, require a plane and smooth surface to provide reliable and meaningful results. The materialographic preparation process described above ensures that the specimen is ideally suited for hardness testing. ATM offers the **Carat 930** for this purpose, a powerful instrument for micro-hardness testing and optical evaluation.

The polished surface in Figure 3 shows several cracks. The straight edge on the left was achieved by milling. The contour of the welded seams is not visible. For a more detailed examination, the contrast was enhanced by etching. The etched surface is shown in Figure 4. It has more cracks and the colored spots indicate over-etched areas close to several cracks due to etchant residues. The welded seams, which have different dimensions, are well visible. The layer-by-layer deposition technique effectuates heat treatment of the subjacent layer. A heat affected zone (HAZ) is formed and causes a change in the microstructure, affecting the specimen’s properties. For example, the hardness may be reduced, resulting in mechanical stress. As layers of different hardness are deposited one on top of the other, the mechanical stress continuously increases and may lead to so-called secondary cracks. A reason for the formation of primary cracks are cooling gradients during deposition. Figure 5 shows a magnification of single welding beads and their corresponding heat affected zones. Hardness testing can reveal the differences in hardness of the deposited layers.