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Identification of Heterogeneous Properties of a Metallic Polycrystal by Stereoscopic Measurements at Microscale

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Résumé — This study focuses on developing algorithms to connect optical and electron microscopy images with macroscopic stress-strain data and micro deformation data via DIC technique and utilize reverse methods to predict the polycrystals' mechanical behavior. Such algorithms and techniques aim to bridge the gap between experimental and numerical analysis by utilizing several constitutive laws. The image processing methods, from manual to automatic ways, allowing the discretization of each grain in a microstructural image, as an intermediate step, plays a crucial role at its finest.

However, challenges remain at the granular scale, where a comprehensive material law is needed to explain individual grain mechanics. To address this, innovative approaches are required to advance our understanding and bridge the existing gap, as discussed in this case.

Mots clés — microstructure characterization, polycrystal material, optical image, plasticity, segmentation.

1 Introduction

At microscale, materials often exhibit discontinuous behavior owing to various defects, including dislocation pile-ups, nanoscale voids, inclusions, grain boundary slidings, and cracks. These defects have a significant impact on material properties, such as strength and toughness [1, 2, 3]. Extensive research in materials science and engineering has thoroughly explored this phenomenon of discontinuous behavior in materials [4]. This research examines how microstructural features like grain size, texture, and defects influence material behavior, offering insights into how these factors can be harnessed to enhance material properties.

Another important phenomenon is that the microstructure of polycrystalline metals is characterized by the presence of grain boundaries, which serve as interfaces between adjacent grains. These grain boundaries exert a significant influence on various material properties, including mechanical strength, thermal stability, and electrical conductivity. Additionally, they offer opportunities for manipulating material properties through techniques like grain boundary engineering or texture control [5]. Thorough research has explored the impact of grain boundaries on the behavior of polycrystalline metals [6].

Under mechanical loading, the behavior of such materials is influenced by a combination of factors, encompassing the orientation of individual grains, grain shape and size and distribution. Researchers have extensively investigated the mechanical behavior of polycrystalline metals, utilizing diverse methods, including experimental techniques, theoretical models, and computer simulations [7, 8]. Therefore, advancements in materials characterization techniques, such as electron microscopy, X-ray diffraction, and atomistic simulations, have delved deeper into the structure and behavior of polycrystalline metals at the atomic scale. These techniques facilitate the visualization and analysis of grain boundaries, defects, and their impacts on material properties [9, 10].

To attain a comprehensive understanding of a material's microstructure, it is essential to employ complementary characterization techniques with stereoscopic measurements. These measurements, commonly employed in characterizing polycrystalline metals, provide valuable insights into the distribution and morphology of grains, as well as grain boundaries and defects [11]. Stereoscopic measurements, offering a unique three-dimensional perspective, complement other techniques, especially in the study of the behavior of polycrystalline materials under deformation, where the interaction between grains and grain boundaries significantly impacts the material's overall mechanical properties [12]. Another pivotal

aspect to address in the context of this study is the phenomenon of strain localization in polycrystalline metals; the mechanical phenomenon wherein plastic deformation becomes concentrated within a confined region or specific set of grains due to the microstructure's heterogeneity, grain boundaries, and defects. This process creates stress concentrations and impedes the spread of plastic deformation, often leading to the formation of shear bands—narrow zones of highly strained material that can result in failure [13, 14].

Numerous experimental techniques have been developed to investigate the mechanisms of strain localization in polycrystalline metals. For instance, in-situ deformation tests, combined with digital image correlation techniques, enable researchers to monitor the deformation of individual grains and pinpoint the locations of strain localization [16]. Utilizing electron backscatter diffraction (EBSD) for microstructural analysis reveals the orientation and morphology of grains and their corresponding boundaries, all of which play a significant role in influencing the onset and progression of localized deformation [17]. Moreover, high-resolution imaging techniques, such as transmission electron microscopy (TEM), offer detailed insights into the dislocation structure and its interaction with grain boundaries ([18]. Regarding constitutive laws for mechanical deformation in crystalline materials, a comprehensive review of existing solutions and research is provided by Roters et al. (2010) [19].

2 Experimental Procedure

The experimental study began with thermal treatment and polishing of 316L stainless steel specimens. The material underwent annealing at 1200°C for three hours, followed by gradual cooling to enhance strength through grain enlargement and reduction of grain boundaries. Subsequently, a polishing procedure was implemented to achieve a surface roughness of 1 micron, and Nitric Acid (HNO₃) was applied to enhance the visibility of grain boundaries.

The microtensile testing apparatus, as shown in Figure 1, was then employed, attached to a Leica M205C stereomicroscope. This optical microscope provided high-resolution images at a magnification of 1280x, forming the basis for subsequent numerical analyses. To further explore the crystallographic structure, a Scanning Electron Microscope (SEM) using the electron backscattering technique was employed, offering finer details such as grain size, orientation, and grain boundaries beyond the capabilities of an optical microscope.

Additionally, optical microscopy coupled with digital image correlation (DIC) technique was utilized to measure microscale deformation during tensile testing. The overall experimental setup is illustrated in Figure 2, showcasing the configurations of the stereomicroscope and the tensile micromachine. In summary, the experimental approach involved a comprehensive examination of the material's mechanical behavior, with a focus on the effects of thermal treatment and polishing, coupled with an in-depth analysis of crystallographic structure and deformation characteristics through advanced microscopy techniques.

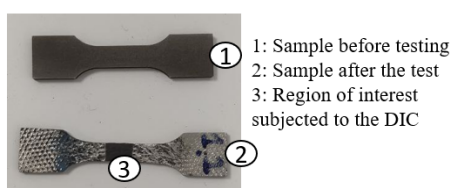


FIGURE 1 – Test samples before and after the tensile test.

3 Processing the Microstructure Image

It is important to remember that one of the main goals in this study is to correlate the numerical and the experimental results of stereoscopic measurements of tensile tests at the microscale. Such images obtained through optical microscopy and electron microscopy before and after the experiments mean not much but a bunch of pixels for a numerical analysis intended (finite element analysis). Thus, there is the need to transform them into meaningful data to proceed with numerical analysis.

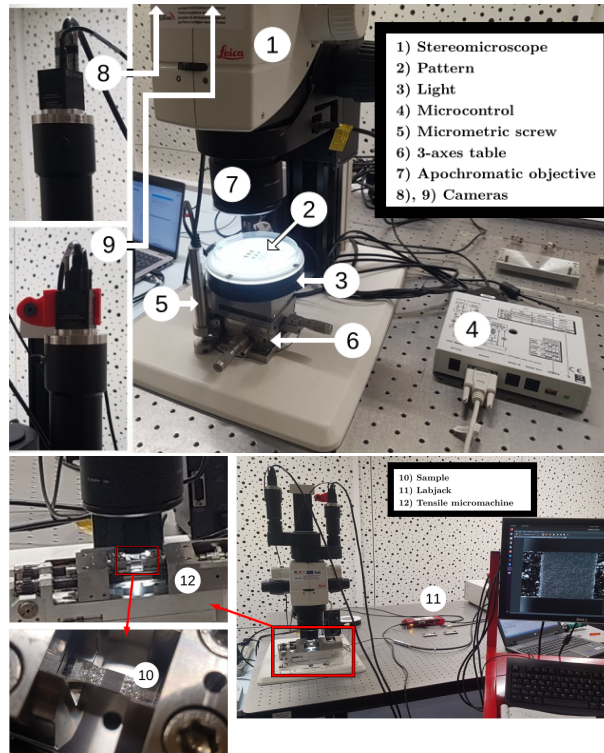


FIGURE 2 – Experimental setup for the stereomicroscope and the tensile micromachine.

The processed image represents the actual microstructure at its finest and becomes an intermediate step between the raw image taken from the optical microscope and the final geometry generated by CristalX software in which then will be subjected to finite element analysis.

3.1 Preprocessing : Initial Arrangements

The meaning behind the preprocessing process in this section is of the manipulations that were applied on the raw optical microscopy image to convert the optical microscopy image into the best possible state. In the preliminary stages of the investigation, the optical microscopy image (Figure 3a) was acquired, capturing the subject under study. Prior to commencing our analyses, the image underwent essential preprocessing steps, which were diligently conducted by Eddy Caron. Specifically, the open-source image editing software, GIMP (GNU Image Manipulation Program), was employed for this purpose [20]. Upon completion of the preprocessing procedures, we obtained Figure 3b, where the image was transformed into a grayscale representation, showcasing distinct grain boundaries at different steps.

During the preprocessing stage, it is essential to highlight three primary challenges that were encountered :

- **Incomplete Grain Boundaries** : Following the electro-chemical treatment of the sample and the preliminary image editing performed using GIMP by Caron, it became evident that further refinements were necessary. A comparative analysis between the Electron Backscatter Diffraction (EBSD) image in Figure 4a and the optical microscopy image in Figure 3b revealed notable discrepancies in the representation of grain boundaries. The grain boundaries in Figure 3b were observed to deviate from reality, displaying a discernible difference in the number of grains when compared to the EBSD reference. Specifically, the optical image exhibited a reduced number of grains compared to the EBSD map. To address this limitation, manual intervention became imperative, entailing the creation of artificial grains. Leveraging the capabilities of GIMP, artificial grains were meticulously drawn, while simultaneously eliminating defects present in Figure 3b, the intermediate image derived from the initial preprocessing steps. Additionally, some unwanted grains and branches emerged as a result of the color conversion to black and white during the image editing process. Consequently, appropriate clearing techniques were applied to address these issues by utilizing the same software. This laborious process resulted in the generation of

the final image (Figure 3c) that would serve as the basis for subsequent processes.

- **Misalignment and Resolution Difference** : Figure 3a displayed a noticeable misalignment of the sample during measurements, necessitating corrective measures. However, it remains uncertain whether the sample was also tilted during the testing phase. As a consequence of the disparity in resolution between the deformation field derived from the digital image correlation technique and its optical microscopy counterpart, a requisite measure was taken to normalize the images to a resolution of 3000x4000 pixels. Figure 3d is the final version of the preprocessing stage before its use in segmentation process.

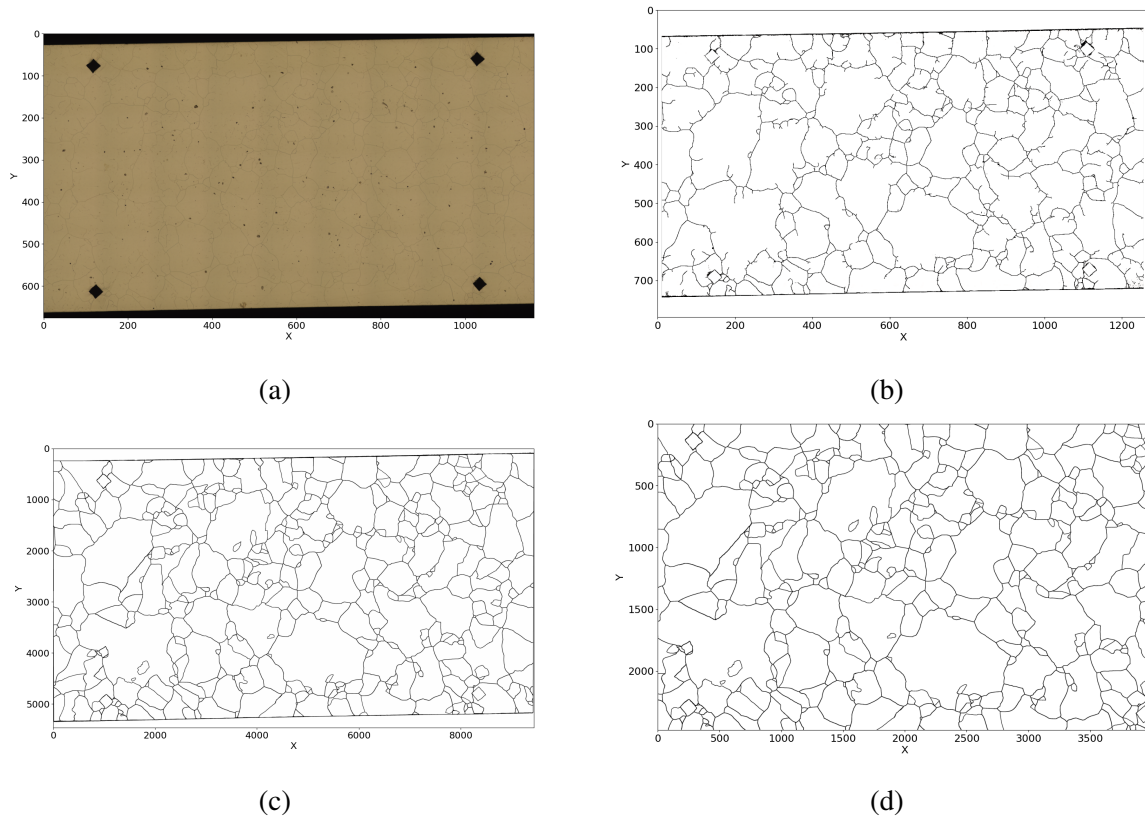


FIGURE 3 – (a) Raw microstructure image taken by optical microscopy, (b) Initially preprocessed microstructure on GIMP, (c) Cleared image by taking into account grain boundary corrections with EBSD map, (d) The final microstructure image to be used in Segmentation process

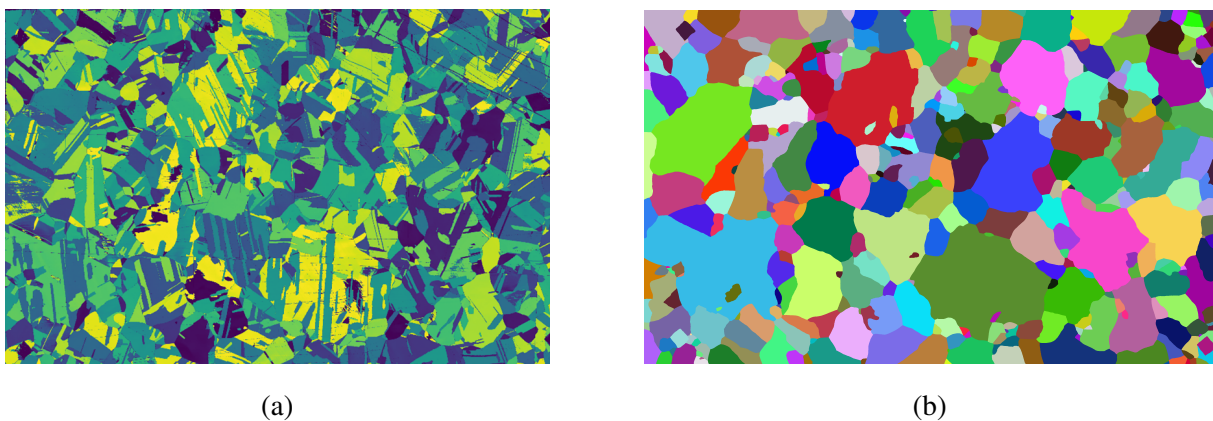


FIGURE 4 – (a) Electron microscopy image of the microstructure with EBSD method, (b) segmented microstructure

3.2 Segmentation of the Microstructure Image

In this context, *segmentation* specifically denotes the algorithmic process of pixel partitioning within an image depicting a metallic microstructure. The resultant segmented image reveals distinct elements, colors, shapes, and various visual characteristics when contrasted with the original image.

Within the scope of this research, the utilization of segmentation is strictly confined to the realm of grain identification. The primary objective of this process lies in the establishment of isolated pixel clusters that represent individual grains. This systematic approach greatly facilitates the extraction of critical microstructural data, including parameters such as grain count, grain size (area), and related quantitative attributes.

Given that the segmentation process heavily relies on heuristic methods and the judicious selection of techniques aimed at producing output images more amenable to subsequent analyses, the success of segmentation depends on the achievement of an output that is better suited for further scrutiny. To this end, the deployment of multiple methods becomes essential, contributing to the determination of the most appropriate approach based on factors such as grain count, grain size, and other relevant parameters."

While detailed discussions of various segmentation methods are reserved for future studies, the current investigation adopts the Voronoi-Otsu thresholding method *originally this* as the designated segmentation algorithm. This approach employs a black and white halo pattern, as depicted in Figure 3. After applying adjustments for size, rotation, and resolution, the thresholding technique is used on the microstructure, resulting in a clear image with distinct grain boundaries.

Moreover, this method assigns a unique index number to the pixels composing each grain. This index assignment facilitates the alignment of strain data from the image for subsequent stages of analysis. (*python for microscopy*). A modified version of *CristalX_m* incorporates this method in its *segmentation.py* module.

3.3 An Automated Transformation Method of Microstructure Images : CristalX

The feasibility of translating microscale experimental testing into the numerical domain has been enhanced through the development of *CristalX*, a semi-automated open-source software in Python by Z. Csati [21]. The geometry generation phase, while promising, introduces maintenance challenges related to software dependencies and geometric imperfections. Manual intervention is required in certain cases, and geometry repair operations are performed within the Salome platform.

Once the geometry in Figure 5 is generated, meshing and repair operations followed after. The absence of gaps between grains is noted. Quality control assesses surface normals and geometric imperfections, indicating the need for future optimization.

In Figure 5, the central colored region represents the defined region of interest in the sample. Surrounding this central area, 'superficial grains,' artificially generated by CristalX, are incorporated. These additional elements help mitigate potential disturbances stemming from far-field boundary conditions that might affect the analysis.

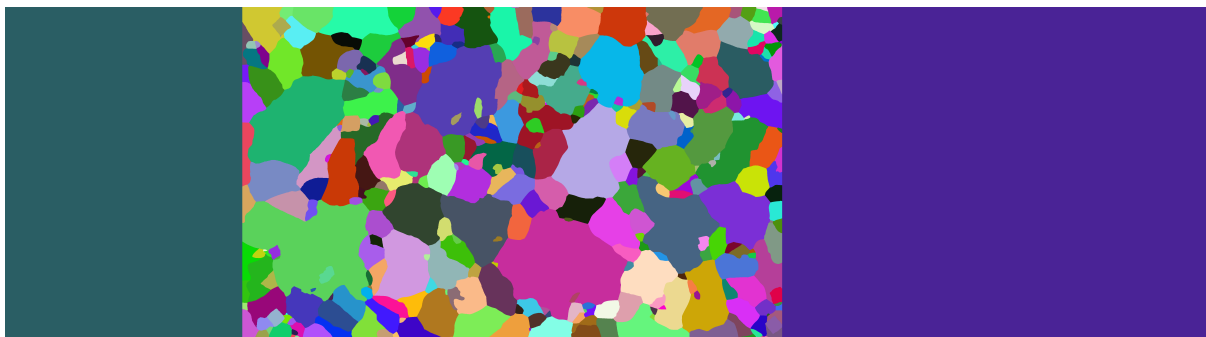


FIGURE 5 – Final microstructure image after segmentation with extended domains

In addition to the inherent segmentation solutions integrated into CristalX, which yield comparable outcomes to various other libraries for labeling grains, as previously stated the post-segmentation phase reveals grains characterized by unique yet randomly assigned indices as previously. Each pixel associated

with a particular grain shares the same index, thereby facilitating the computation of estimated grain parameters, including area, shape, and size as illustrated in Figure 6.

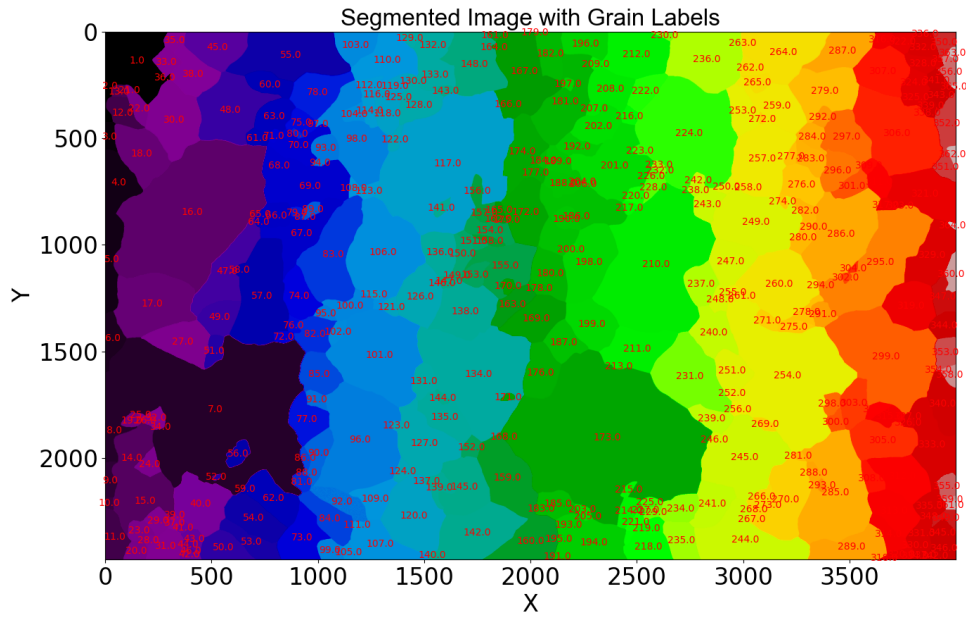


FIGURE 6 – Segmented image with unique grain labels assigned

4 Conclusion and Discussion

In conclusion, upon completion of tests, observations are made through optical and scanning electron microscopes, and necessary image processing applications, including segmentation for grain identification, are performed. Utilizing the open-source software CrystalX, the closest representative geometry is generated with manual interventions.

The subsequent phase of the study involves determining material behavior laws best suited for this case and performing numerical analyses to establish a coupling between numerical simulations and the experimental conditions of the test. This progression is integral for a comprehensive understanding of the material's behavior.

Throughout the investigation, procedures are meticulously traced from the raw material's initial state to its digital transformation. The accuracy of visual operations on the microstructure depends on the identification of grains within the geometric framework generated by CrystalX. The proficiency of the algorithm in grain detection during segmentation is pivotal, shaping subsequent processing steps.

Additionally, the effectiveness of the thermal process in outlining grain boundaries on the object's surface is crucial, influencing the selection of precise processes in later stages. The quality of the optical microscope's acquired image, influenced by factors such as illumination and magnification, plays a key role in deciding on diverse image processing techniques in subsequent phases.

Following the refinement of resulting geometries and defect removal, the next critical step involves organizing data from tensile tests or relevant material information tailored to each grain. This process is essential for grain mapping and location determination, thus, for numerical analysis, marking a crucial juncture in the investigative journey.

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