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# Study of the effect of Accumulative Angular Drawing deformation route on grain refinement in 304L stainless steel

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## Abstract

Stainless steel grades – both austenitic and ferritic – find a wide critical applications due to their superior properties such as high strength, excellent corrosion resistance and good biocompatibility. Manufacturing e.g. medical equipment and implants made of stainless steel grades often involves wire drawing processes. Further improvement of mechanical properties requires more research in the field of grain refinement, which is the main strengthening mechanism, that does not decrease the ductility and toughness. In the present work, capabilities of grain refinement in 304 austenitic stainless steel using recently developed technology of Accumulative Angular Drawing (AAD) was studied. Focus was put on the analysis of the influence of process parameters on inhomogeneity of microstructure and related mechanical properties. The AAD process uses the effect of complex deformation mode and strong strain accumulation to obtain a controlled level of heterogeneity of the microstructure and the resulting properties. In the current work, wire rods of the 304L stainless steel were drawn using linear, stepped and cranked drawing dies arrangements. The main goal of this work was aimed at understanding the effect of deformation conditions on the microstructure and properties of 304 stainless steel drawn wires, by studying the mechanical properties after drawing (hardness, tensile strength) and microstructure evolution during the AAD process. The effects of complex deformation modes and strong strain accumulation were studied in the light of possible grain refinement and mechanical properties.

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*Keywords:* Stainless steel; Accumulative Angular Drawing, Severe Plastic Deformation effects

## 1. Introduction

The world production of stainless steels is constantly growing. Nowadays, new applications of face centered cubic (FCC) structure materials are found in many branches of industry such as petrochemical, food or medicine. Stainless steels are used because their biggest advantage is the corrosion resistance. An important feature of these steels is their high ductility and elasticity, compared to non-alloyed steel counterparts. To obtain the desired properties of steel, its

chemical composition is controlled by adding appropriate alloying elements. To increase the strength of stainless steels, chromium, nickel, tungsten, molybdenum and vanadium are added [1]. In addition to interference in the chemical compositions or heat treatment, plastic deformation increases strength as well. The wire drawing process of stainless steels is widely used in the industrial field of applications such as automotive and electrical components [2]. Drawn elements are used in many types of fasteners [3]. The cross-section area during drawing process is reduced by plastic deformation

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through a drawing die. One of most important process parameters are the reduction area value, die angle and strain rate. Characteristic feature of the process is the strain inhomogeneity in the cross-section of the drawn wires [4]. The level of strain heterogeneity leads to the modifications of the microstructure and properties in the cross-section [5]. That in turn, leads to improvement the conventional drawing processes to achieve a special microstructure-properties combination.

The Accumulative Angular Drawing (AAD) is a recently developed technique which is a new powerful tool that allow to produce large quantities of wires with enhanced properties and microstructure. This kind of method is characterized by a complex strain path history resulting from various deformation modes (Fig. 1a, 1b, 1c). The AAD process induces high strain accumulation in the surface layers of the wire. That allows to obtain increased mechanical properties. Similarly, to the Serve Plastic Deformation processes, the large strain accumulation is introduced to refine the microstructure of a drawn wires [6-9].

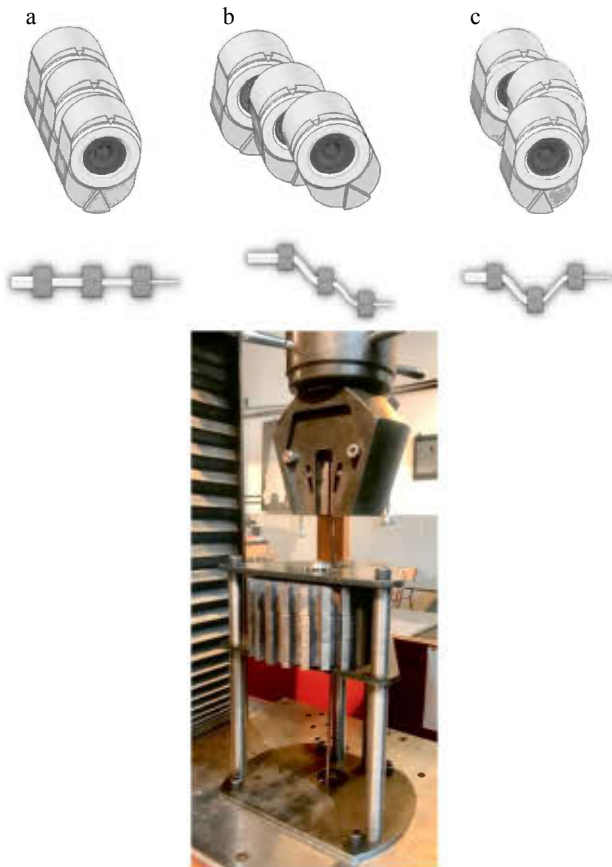


Fig. 1. Drawing dies arrangements: (a) linear; (b) stepped; (c) cranked; (d) installed AAD system on Instron 1196 frame.

## 2. Experimental methods

The AAD process uses a high strain accumulation in the surface area which allows one to achieve grain refinement and increased strength in a wire. In the present study wires made from 304 austenitic stainless steel with the composition and properties

showed in Table 1, were investigated. The initial wire-rod in the as-hot rolled conditions is characterized by coarse equiaxed grains and annealing twins with relatively weak texture (Fig. 2, 3). In the current work, the wire rods with diameter of 5.5 mm were drawn by AAD process to obtain a final diameter of 4.0mm by applying linear, stepped and cranked arrangement of drawing dies (Fig. 1a, 1b, 1c).

Table 1. Chemical composition and properties as received 304 SS wires.

Thick (diameter) mm	Chemical composition (wt%, x1000)										Mechanical properties		
	C	Si	Mn	P	S	Ni	Cr	Mo	Cu	N	TS N/mm <sup>2</sup>	EL %	HV
5.50	34	34	182	29	0.7	803	1823	14	70	308	585	69	156

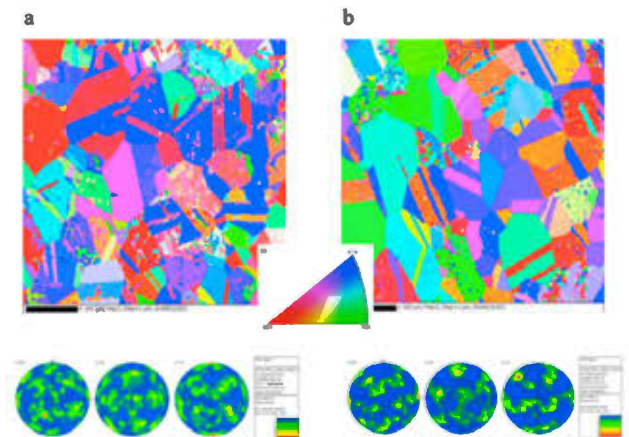


Fig. 2. EBSD maps and pole figures of initial material: (a) transverse cross-section; (b) longitudinal cross-section.

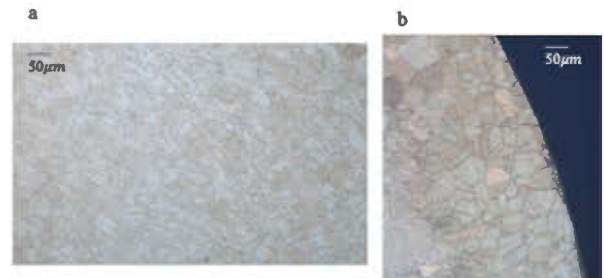


Fig. 3. Etched microstructures acquired using optical microscope: (a) middle of transverse cross section; (b) surface area.

To ensure proper surface preparation, before the drawing a lubricant layer was applied on the surface of the wires. It was done in two steps, first by immersing the wires in a hot aqueous salt solution to obtain a deposited calcium chloride layer on the wire surface. Then, the lubrication during the process was performed using mixture of mineral oil and sodium calcium soap powder. Based on the calculations, the appropriate drawing dimensions and drawing pattern were determined (Fig. 1a, 1b, 1c). In the stepped arrangement of the dies, the AAD drawing process was realized with diameter reductions of 4.8, 4.3 and 4.0 mm whereas in the case of linear and stepped arrangements the following diameter reductions were realized in the subsequent passes: 5.0, 4.8, 4.6 + 4.0 mm calibration for cranked drawing path. Accordingly, the deformations

(equivalent strain) during the process were 0.23 (first drawing die), 0.19 (second drawing die) and 0.13 (third drawing die). The AAD process was performed with 20 mm/min traverse speed of Instron 1196 testing machine, where the whole system was installed (Fig. 1d) and forces during process were measured (Fig. 7). In the current, initial work 3 different dies misalignment angles were analyzed – namely 7, 10 and 20 deg. Additionally, for reference and comparison, linear drawing was also performed with the same diameter reductions and similar speed.

The aim of the work was to assess the possibilities of grain refinement and inhomogeneity of both microstructure and properties as an effect of applied process parameters. For that purpose, specimens for microstructure study were cut from the drawn wires, both in the longitudinal and transverse directions. The sample preparation for optical microscope analysis was done by grinding specimens down to 2500 SiC water papers, then by polishing with diamond suspensions (6, 3 and 1 microns respectively). To reveal the microstructure, the electroetching was performed using 60% nitric acid solution. To determine the mechanical properties of both the initial and deformed materials, the Vickers hardness measurements (HV0.5) and tensile tests were performed.

### 3. Results

The microstructures obtained using optical microscope are presented in Fig. 4. It can be seen that the application of the AAD drawing results in the inhomogeneity of grain refinement on the cross section. Looking at the initial material, the grain size in the core and at the surface area was uniform and on average equal to 100 microns (measured by linear intercept method). The hardness of the initial wire averaged at the level of 145 HV0.5 (more accurate locations and values are shown in Fig. 5a). Analyzing the results of the linear drawing process, it can be noted that a definite grain refinement was obtained compared to the initial material. The measurement of the surface area showed stronger grain refinement - to the level of 48.5 microns, whereas in the middle of the cross-section the grain size was around 64 microns (Fig. 4b). This behavior is typical to conventional wire drawing processes. In the AAD-drawn wires stronger grain refinement was obtained. The minimum grain size (around 34 microns) was observed in the specimen drawn in the stepped manner with the higher dies misalignment angle (Fig. 5c, 5d). In the case of cranked drawing arrangement, a significant increase in strength properties and grain refinement, up to around 420 and 440 HV0.5 (respectively for crank and cranked calibrated) is also noticeable. All hardness measurement results showed significant increase in hardness. Recalling, in undeformed material, the hardness was 145 HV0.5 and in linearly drawn material it reached 438 HV0.5 (average perimeter, surface area). In the AAD - drawn wires (with 7 and 10 deg of dies misalignment) further increase in hardness was observed. The cross-section of an angle wire drawn at 7 degrees showed hardness of 440 HV0.5 while at 10 degrees the hardness was 450 HV0.5. (Fig. 5c, 5d). Comparing the values for angular (stepped and cranked) the level of hardness increase is similar. For linear and angular drawing, surface grain refinement with respect to the core is visible. Linear drawing is characterized by a greater degree of fragmentation on the cross-section (27%) but the surface areas on angularly drawn sections are more

fragmented. This is the effect of additional burnishing during angular drawing. However, when compared to the core, the degree of fragmentation is smaller, 20% and 17%, respectively, for angles of 7 and 10 degrees. The larger the angle, the greater the burnishing and hardening of the wire at the surface, which is why the average difference in grain size in this area is only 1 micron, while the difference in hardness is already 10 HV0.5. It can therefore be concluded that increasing the torsion angle between the consecutive drawing dies - and thus increasing the deformation energy - higher strengthening effect in the drawn product is observed. The above dependence of strengthening with increasing torsion angle is confirmed on the Fig. 6; the deformation resistance as the angle increases, so similar hypothesis can be put forward here that higher material strengthening occurs with more sophisticated deformation path.

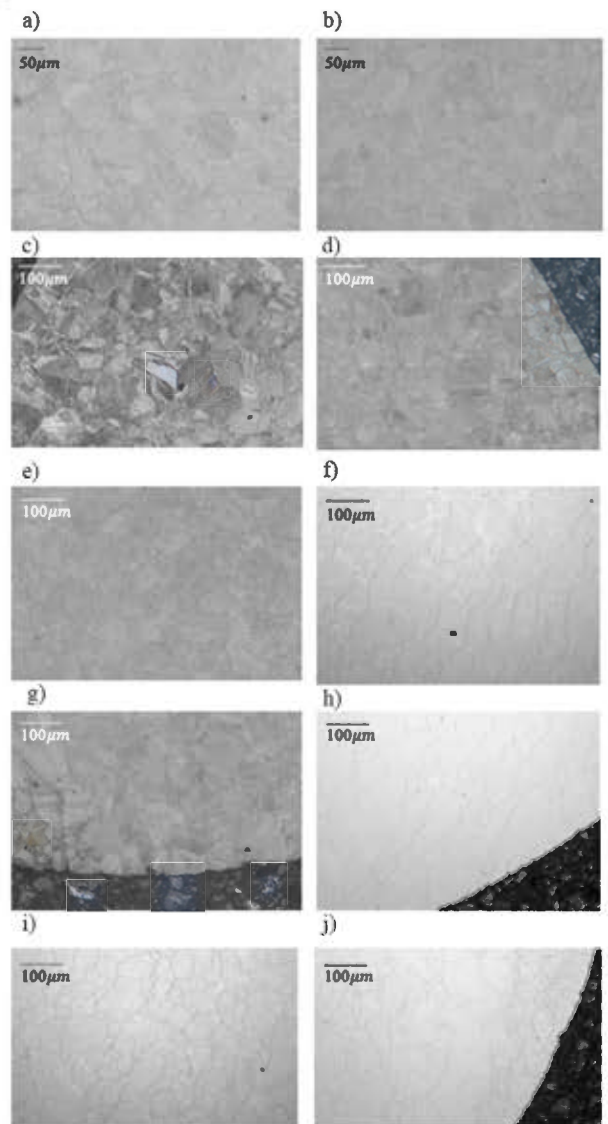


Fig. 4. (a, c) Linear and (b, d-j) angular (both misalignment of 7, 10, 20 deg.) microstructures (mag x20): (a) linear middle, (c) linear surface, (b) AAD stepped 7 deg. middle, (d) AAD stepped 7 deg. surface, (e) AAD stepped 10 deg. middle, (g) AAD stepped 10 deg. surface, (f) AAD cranked 20 deg. middle, (h) AAD cranked 20 deg. surface, (i) AAD cranked 20 deg. calibrated middle, (j) AAD cranked 20 deg. calibrated surface.

To assess the effect of applied process conditions on the inhomogeneity of microstructure and properties, tensile tests were performed. First of all, the tensile tests were performed on initial wire-rod material to identify the mechanical properties i.e. yield strength (YS), Ultimate Tensile Strength (UTS) and YS/UTS ratio) as well as ductility (Fig. 6). The UTS of initial 304 stainless steel wire was 630 MPa. After drawing UTS increased to respectively 1460 MPa, 1475 MPa, 1463 MPa and 1405 MPa for linear, AAD stepped 7 deg. and AAD stepped 10 deg., AAD cranked 20 deg. (+calibration 4.0) deformation paths.

As it is shown in Fig. 5, the initial wire-rod was characterized by YS of 250 MPa. The two others curves – after linear drawing and stepped arrangement – show that the drawn wires have significantly hardened. The YS/UTS ratio in the initial wire no more than 0,4 but in case of all drawn wires (linear and angular and angular dies positionings), YS/UTS ratios are 0,99 and UTS itself is 1460, 1463 and 1475 MPa, for linear and angular drawing, respectively.

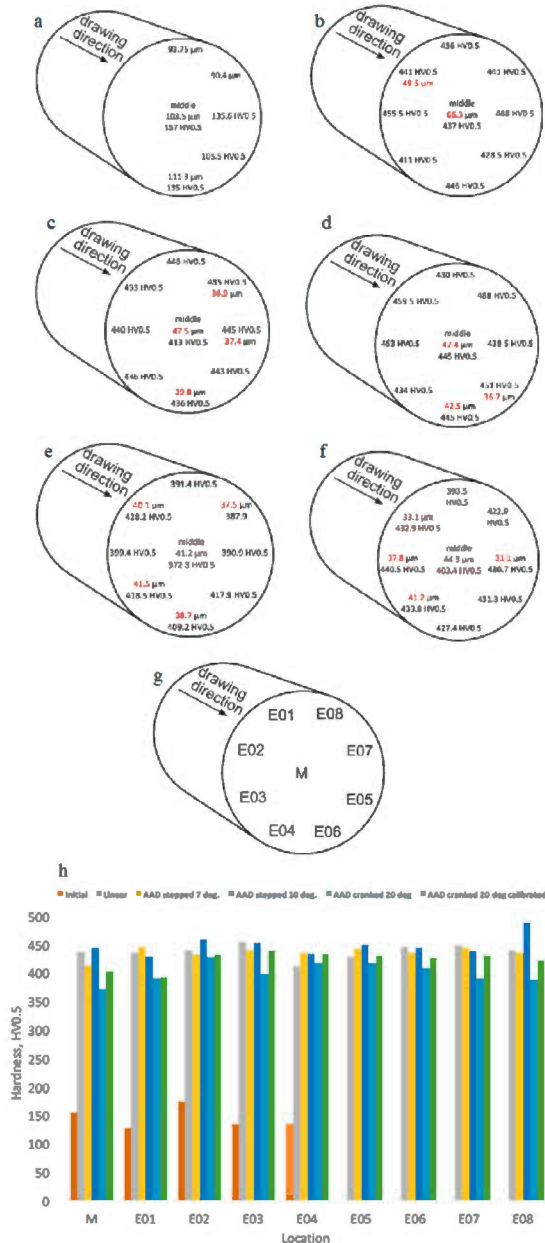


Fig. 5. Grain size and Vickers (HV0.5) results on the transverse cross-section: (a) initial; (b) linear; (c) AAD stepped 7 deg.; (d) AAD stepped 10 deg.; (e) AAD cranked 20 deg.; (f) AAD cranked 20 deg. calibrated.; (g) measuring locations; (h) Vickers values of all samples.

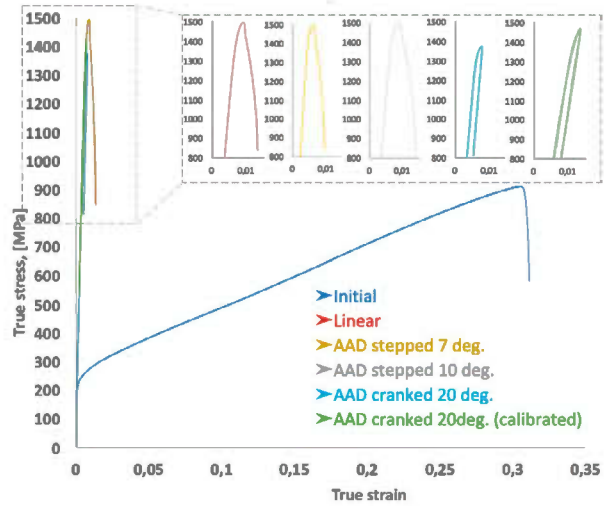


Fig. 6. True strain-stress curves obtained on studied wires using quasi-static tensile test.

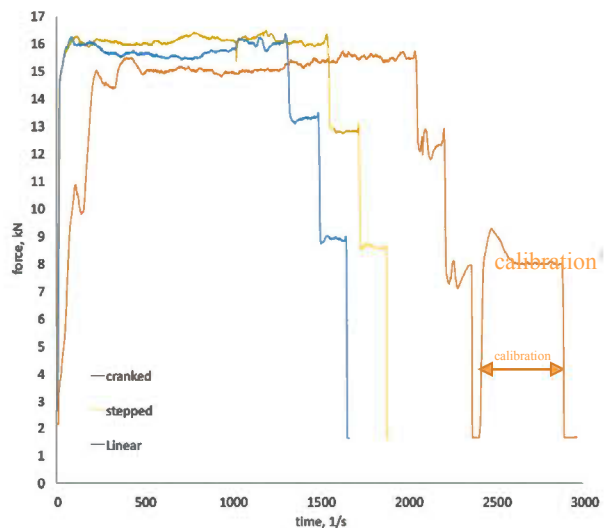


Fig. 7. Forces measured during the AAD process.

Performed tensile tests have shown that the initial wire rod is characterized by a very high ductility (uniform elongation). However, after drawing, drastic drop in ductility was observed what resulted from significant level of hardening. Minimal differences in the mechanical properties were observed between linear and accumulative angular drawing process – which is contrary to the previously observed results for BCC materials [6]. It may result from the lower number of slip systems in FCC materials, compared to BCC. Also, by applying little number of drawing passes and high diameter

reductions per drawing pass, the AAD effects were not sufficient. The most visible difference is shown in the Fig. 5, where cranked drawing arrangement is characterized by malleable (low HV0.5 values) and near-area strengthened properties. Further work is required where more deformation passes should be introduced with higher misalignment angles between the consecutive drawing dies in order to impose more shear into the deformation mode.

#### 4. Conclusions

In the current work, Accumulative Angular Drawing (AAD) method was applied to 304 stainless steel and the effects of different process parameters on both microstructure and mechanical properties were studied. In this preliminary work it was proved that AAD provides more grain refinement – compared to traditional drawing methods. However the beneficial effects that AAD process offers were poorly visible in the studied materials and hence further work is needed with modified deformation schedules.

#### Acknowledgement

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