

Tensile test and SAXS/WAXS measurements of age hardened 7075 aluminum alloy and low density polyethylene plastic

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Abstract

This report investigates two materials, 7075 aluminum alloy and injection molded low density polyethylene (LDPE) plastic. Tensile tests as well as WAXS and SAXS measurements were performed for both materials. The Al 7075 was age hardened at 160 °C for two hours, causing precipitated particles to form. The precipitates are believed to consist of mainly MgZn₂, which was detected in WAXS. The typical size of the particles was found to be 8.7 nm using SAXS. Aging increased the ultimate tensile stress for Al 7075 by 37 MPa compared to an unaged piece, at the cost of a lower strain at fracture. The LDPE plastic was found to be stronger but less ductile in the direction of the injection molding flow direction, compared to the perpendicular direction. This is explained by a directionality of the microstructure observed in both WAXS and SAXS measurements.

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1 Introduction

In this work, we study several material properties. First, tensile tests are used to investigate the effect of age hardening of the 7075 aluminum alloy as well as how the flow direction of injection molded low density polyethylene (LDPE) affects its strength in tension. Secondly, small and wide angle X-ray scattering (SAXS and WAXS) are used to measure the size of the precipitated particles in Al 7075 and the nanoscale structure of LDPE. Figures 1 and 2 show an overview of the different scales of structures studied for the aluminum alloy and LDPE respectively.

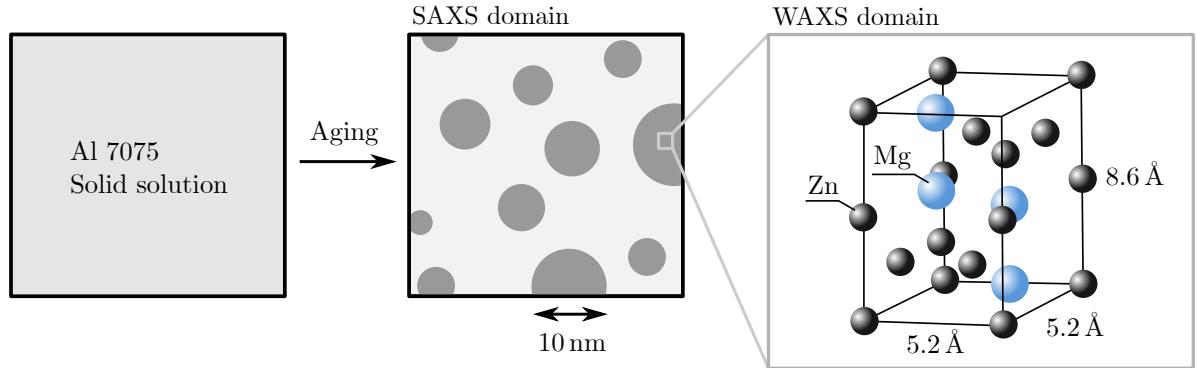


Figure 1. Schematic overview of the nanoscale features studied in precipitation hardened Al 7075 alloy. A solid solution alloy is age hardened to form precipitated particles with a size on the order of ten nanometers. The size is measured more precisely using SAXS. The precipitates are believed to consist of mainly MgZn₂ (hexagonal close packed), for which we found diffraction peaks in WAXS.

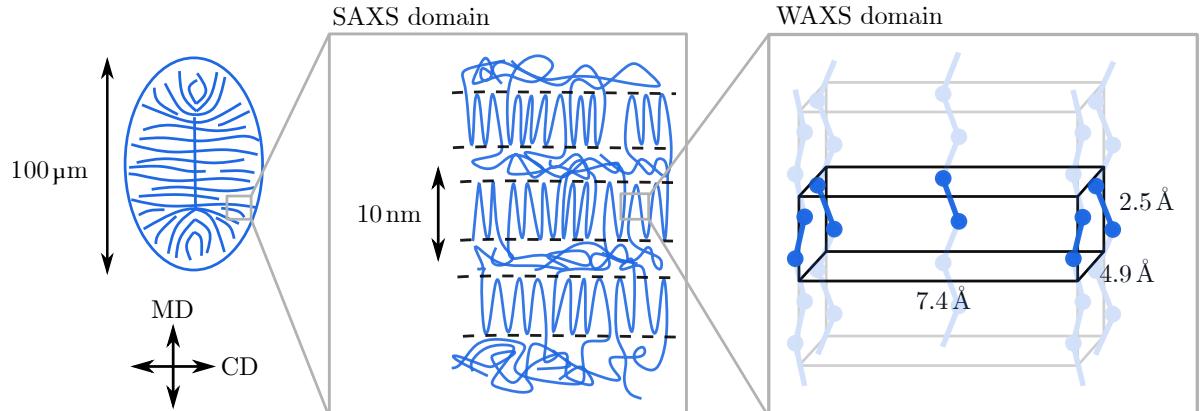


Figure 2. Schematic overview of the nanoscale features studied in LDPE plastic. Elongated spherulites and shish-kebab structures (not shown) form with a direction determined by the flow direction during the injection molding. The spherulites contain crystalline lamella with a thickness on the order of ten nanometers, making them visible in SAXS. The crystalline structure of the lamella results in diffraction peaks that are studied using WAXS. Figure inspired by the lab PM.

2 Method

Tensile tests, WAXS and SAXS measurements were carried out at Chalmers Materials Analysis Laboratory. The LDPE samples were provided by Tetra Pak. In this section, the process of age hardening the Al 7075 is first described as well as the preparation of the LDPE polymer samples. Then, the analysis of the tensile test data is presented along with some equations to estimate the increase in ultimate tensile strength due to the age hardening. Finally, the analysis of the SAXS and WAXS data is described, where the particle size of precipitates and crystalline lamella are estimated and peaks in the WAXS spectra are identified.

2.1 Preparation and measurement of Al 7075

Samples of Al 7075 metal for tensile and diffraction measurements, assumed to be unaged, were provided. The samples were then age hardened in a household oven. The oven was turned on one hour before the heat treatment in order to reach a stable temperature. The temperature was adjusted with the oven knob until the temperature inside, measured with an oven thermometer, was 160 °C. Surprisingly, this occurred when the knob was set to somewhere between 125 °C and 150 °C. The alloy were then placed inside the oven on a flat baking tray for (120 ± 1) min. The measured temperature inside the oven during the age hardening is shown in Figure 3. The temperature oscillated¹ more or less between 150 °C and 170 °C with a period of about 12 min. The average temperature seems to be close to 160 °C.

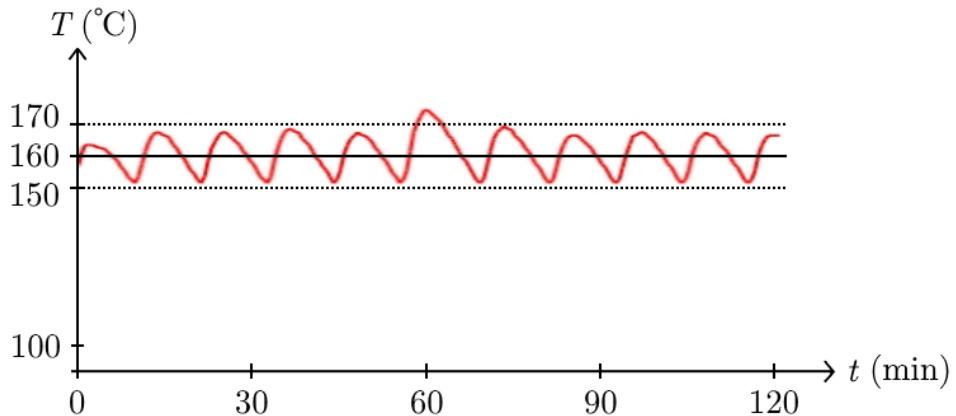


Figure 3. Temperature as function of time from the start of the age hardening, obtained from the built-in temperature logging software of the oven thermometer. Although the oven was set to a fixed temperature, it oscillated between 150 °C and 170 °C (dotted lines) with a period of about 12 min. The average temperature was about 160 °C (solid line).

Tensile tests were performed on both an aged and an unaged sample of Al 7075. Different samples, but with equal amounts of aging, were used for SAXS and WAXS measurements. These samples were not deformed.

2.2 Preparation and measurement of LDPE

Injection molded samples of LDPE for tensile measurements were provided. The samples were cut from a rectangular sheet in two different directions, in the cross direction (CD), perpendicular to the flow direction, and in the machine direction (MD), parallel to the flow direction. This is illustrated in Figure 4. Multiple tensile tests were performed for both MD and CD to reduce the

¹The oscillations are likely due to the use of a "bang-bang" controller, which is either fully turned on or off. This type of regulator is simple and is mainly used in situations where an accurate regulation is not critical.

effects of individual differences between the samples. One MD and one CD sample was studied using SAXS and WAXS. For each sample, both a deformed and relatively undeformed region of the sample was studied.

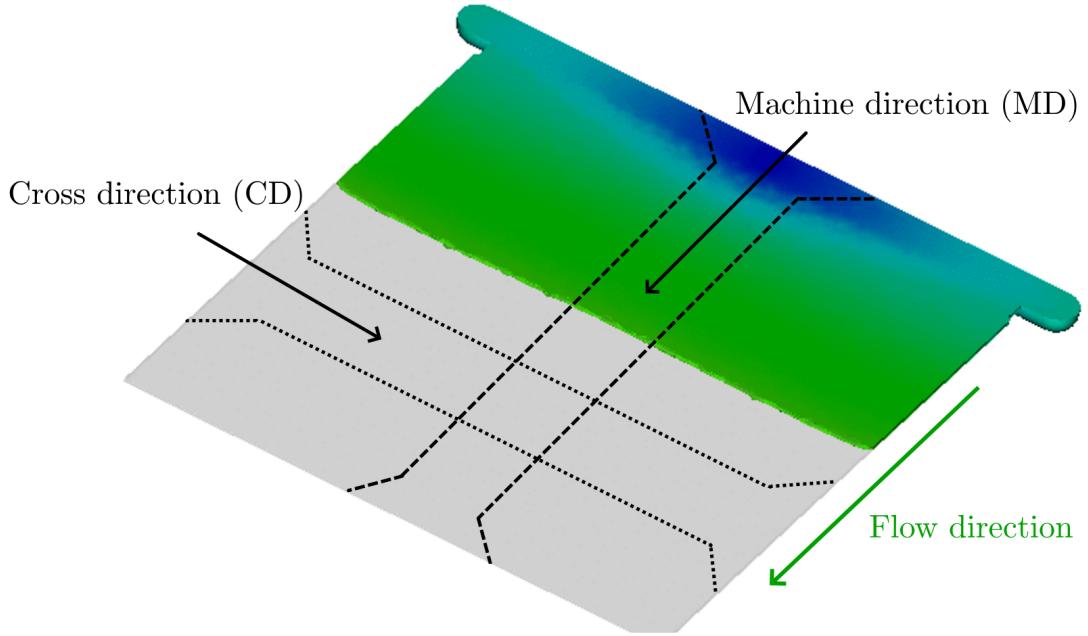


Figure 4. The LDPE were produced by injecting plastic into a mold. The direction of the flow is indicated, as well as the directions in which the LDPE was cut. The machine direction (MD) is parallel to the flow direction and the cross direction (CD) is perpendicular to it.

2.3 Analysis of tensile data

The provided tensile data was given as strain as a function of load force. The tensile stress was calculated as the force divided by the initial cross-sectional area. The ultimate stress and the strain at fracture were taken to be the maximum stress and strain measured, respectively.

For the Al 7075, the expected tensile strength increase from hardening can be estimated using various theoretical formulas. The increase in strength $\Delta\sigma$ due to dislocations bending around smaller precipitated particles is estimated by the Orowan equation

$$\Delta\sigma = \frac{MGb}{L - 2r} \quad (1)$$

where M is the Taylor factor (about 2.5), G is the shear (Young's) modulus, b is the Burgers vector, L is the distance between particles and r is the radius of the particles. If instead the dislocations shear through particles, which is likely to occur for larger particles, the strength increase is expected to be

$$\Delta\sigma = \frac{2M}{r} \sqrt{\frac{\gamma^3 f}{Gb}} \quad (2)$$

where γ is the particle surface energy (typically 20 mJ m^{-2}) and f is the volume fraction of the precipitated particles. Another way to estimate the increase in strength is using

$$\Delta\sigma = \alpha M G b \sqrt{2 N r} \quad (3)$$

where α is usually taken as 0.15 and N is the number density of precipitated particles. Since aluminum has an FCC structure, $\{111\}$ are the most densely packed planes. From this we get

that the Burger vector is $b = a/2 = 2.02 \text{ \AA}$ for aluminum [1]. Also, the shear modulus for Al 7075 is $G = 26.9 \text{ GPa}$ [2].

The distance L between precipitated particles was roughly estimated from the composition of Al 7075. The alloy consists of about 90 wt% aluminum [2]. Assuming that the precipitate and the aluminum has roughly equal density, and that all elements that are not aluminum gather in the precipitates, the volume fraction of precipitate should be at most $f = 0.10$. Assuming that there is one spherical precipitate particle in each cubic volume of side length L , we obtain the interparticle distance

$$L = d \left(\frac{\pi}{6f} \right)^{1/3} \quad (4)$$

where d is the particle diameter estimated from SAXS. The number density is then given by $N = 1/L^3$.

2.4 Analysis of SAXS and WAXS data

In scattering experiments, such as SAXS and WAXS, the momentum transfer vector \vec{q} is the difference between the incoming and the scattered wave vector, $\vec{q} = \vec{k}_0 - \vec{k}_1$. According to Bragg's law, a distance d in a periodically repeating structure, such as atomic planes in a crystal, is related to the momentum transfer $q = |\vec{q}|$ according to

$$d = \frac{2\pi}{q}.$$

The same relation can be used to approximate the size of features without long range order, such as precipitated particles, in a small angle scattering experiment. This allows us to determine the size of structures in a material from the SAXS and WAXS spectrum.

All our SAXS spectra contained a peak corresponding to a characteristic size of some feature. The position of a peak was determined by fitting a Gaussian function to the intensity after subtracting the "background" intensity. The background intensity was obtained by fitting an exponential function in log-log scale to the raw intensity data for high and low q . This is a rather crude method but is considered to be sufficient in order to obtain a rough estimate of the feature size.

In order to identify peaks in the WAXS spectra, theoretical peak positions of various crystal planes expected to be present in the samples were computed until a match was found. For the aluminum sample, the diffraction peaks of MgZn₂ were computed from

$$q = 2\pi \sqrt{\frac{4}{3} \left(\frac{h^2 + hk + k^2}{a^2} \right) + \frac{l^2}{c^2}} \quad (5)$$

where $(hkil)$ are Miller indices for the hexagonal unit cell and $a = 5.220 \text{ \AA}$ and $c = 8.566 \text{ \AA}$ are tabulated lattice parameters for MgZn₂ [3]. For crystalline polyethylene (tetragonal), we used

$$q = 2\pi \sqrt{\left(\frac{h}{a} \right)^2 + \left(\frac{k}{b} \right)^2 + \left(\frac{l}{c} \right)^2} \quad (6)$$

where $a = 7.414 \text{ \AA}$, $b = 4.942 \text{ \AA}$, $c = 2.5473 \text{ \AA}$ are the lattice parameters according to the lab PM. The same formula was used for rutile, a mineral of TiO₂ (a white pigment), but in this case using the tabulated values $a = b = 4.58 \text{ \AA}$, $c = 2.95 \text{ \AA}$.

3 Results

3.1 Tensile tests for Al 7075

Figure 5 shows the stress versus strain curves of the Al 7075 alloy before and after age hardening. Heat treatment made the sample stronger with higher maximum stress, but less ductile with a lower strain at fracture.

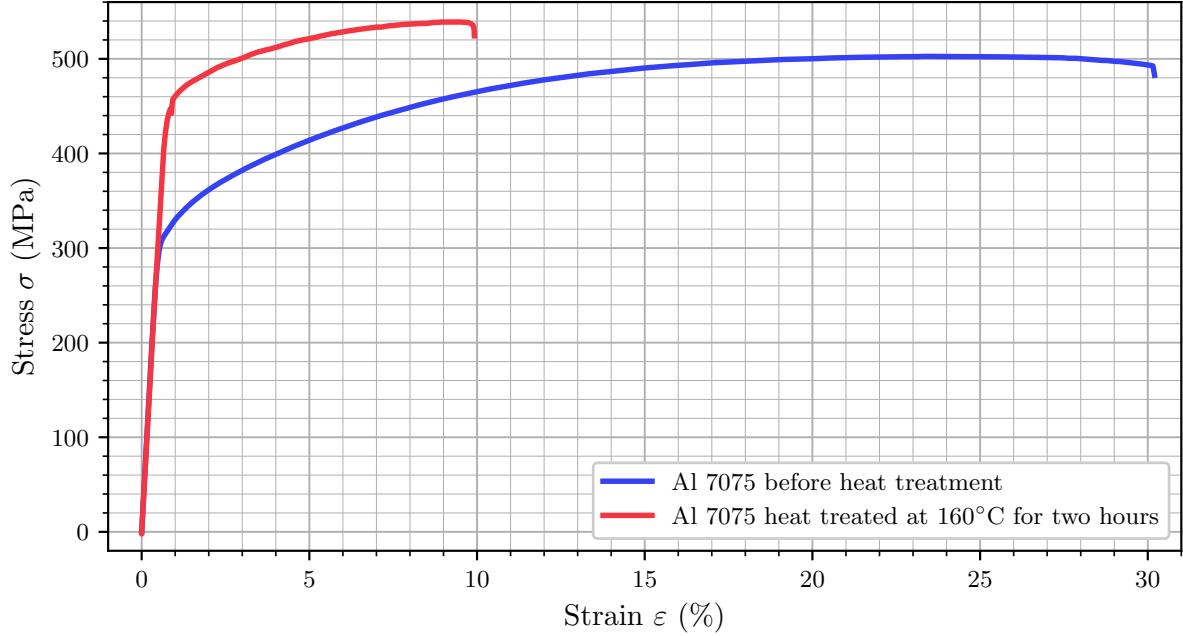


Figure 5. Tensile test of two samples of Al 7075, one before and one after age hardening.

Table 1 shows tensile data for the alloy before and after age hardening. The age hardening increased the ultimate stress with $\Delta\sigma_{UTS} = 37 \text{ MPa}$. The theoretical increase in strength due to age hardening was 2120 MPa, 0.441 MPa, and 102 MPa calculated using (1), (2), and (3), respectively.

Table 1. The stress $\sigma_{0.2}$ at the point of 0.2 % elongation (proof strength), ultimate tensile strength σ_{UTS} , and the strain ε_f at fracture for the Al 7075 alloy before and after age hardening.

Age hardened	$\sigma_{0.2}$ (MPa)	σ_{UTS} (MPa)	ε_f (%)
No	130	502	30
Yes	128	539	10

3.2 SAXS and WAXS measurements for Al 7075

The radially integrated SAXS spectrum for aged Al 7075 is shown in Figure 6. The peak in the spectrum corresponds to a feature in the sample with a typical size of roughly 8.7 nm, believed to be the diameter of the precipitated particles. The peak is broad, which may indicate a wide distribution of particle sizes.

The radially integrated WAXS spectrum for the aged aluminum sample is shown in Figure 7, together with expected peak positions for crystal planes in MgZn₂. The precipitation process may be complex with many intermediate steps, and it is likely that the precipitated particles

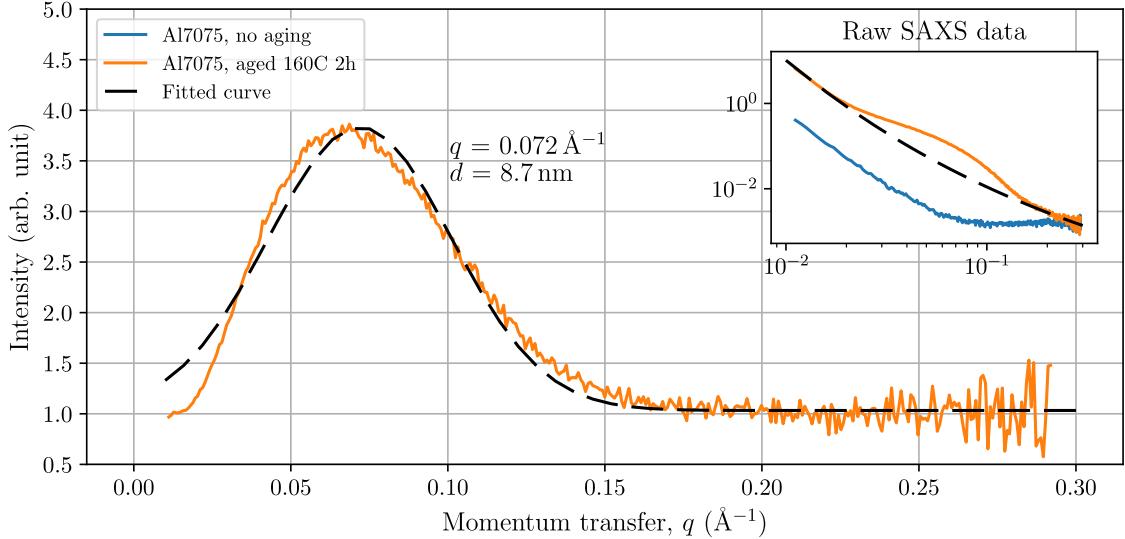


Figure 6. SAXS spectrum for Al 7075 after age hardening with background subtracted. The inset shows raw intensity data with and without aging as well as the fitted curve for the background. The typical size of the precipitated particles are obtained from the peak as $d = 8.7 \text{ nm}$.

contain a range of different crystal structures. This spectrum shows that at least some parts of the sample are MgZn_2 . Diffraction peaks from aluminum are outside the measurement range.

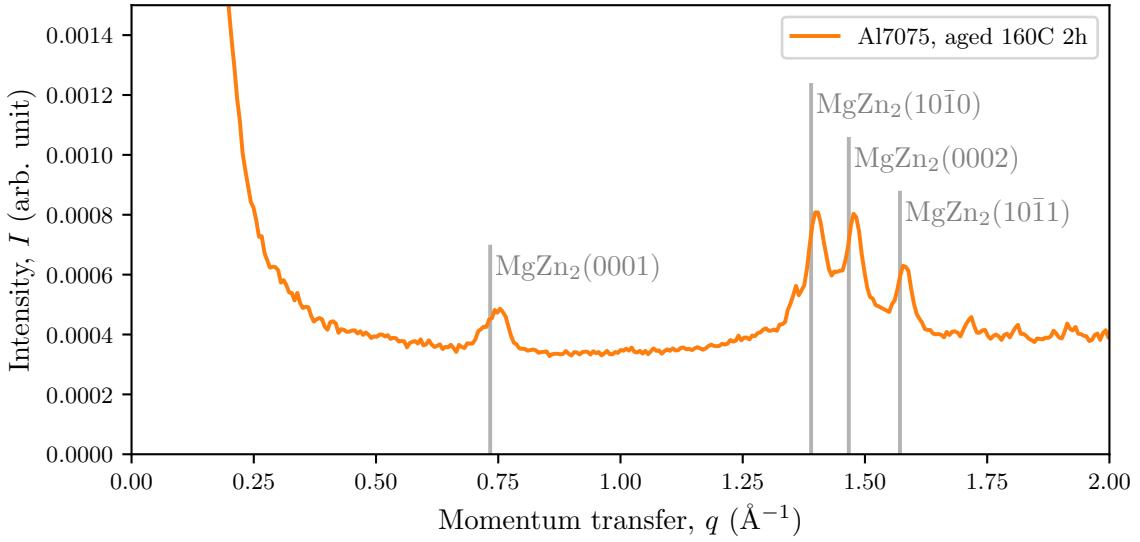
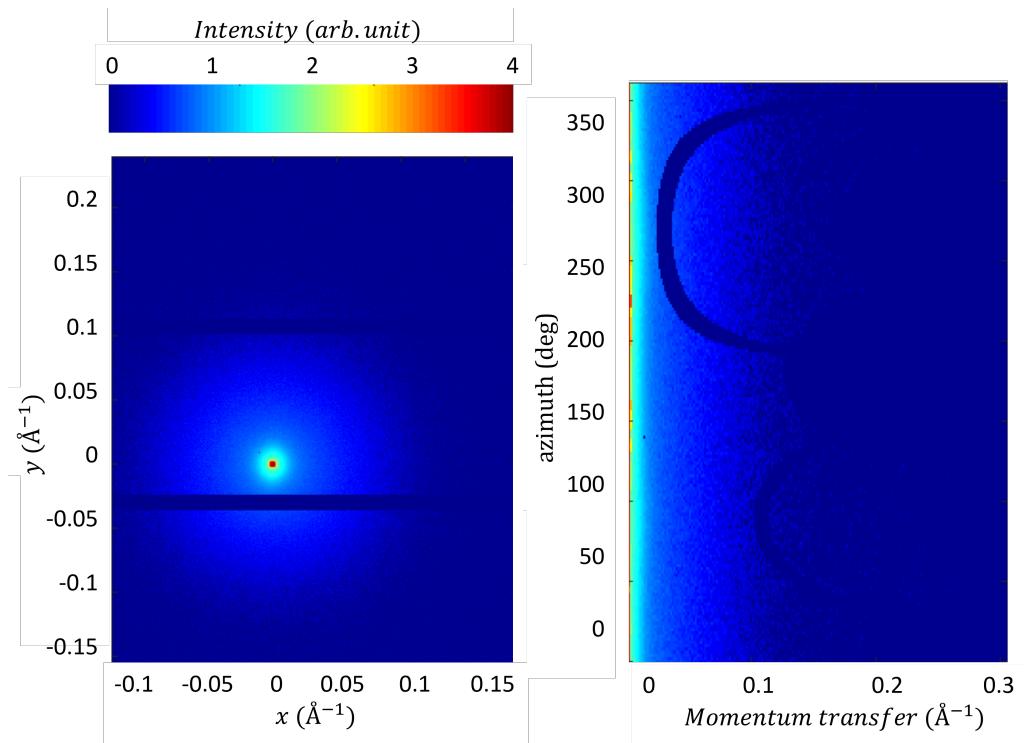
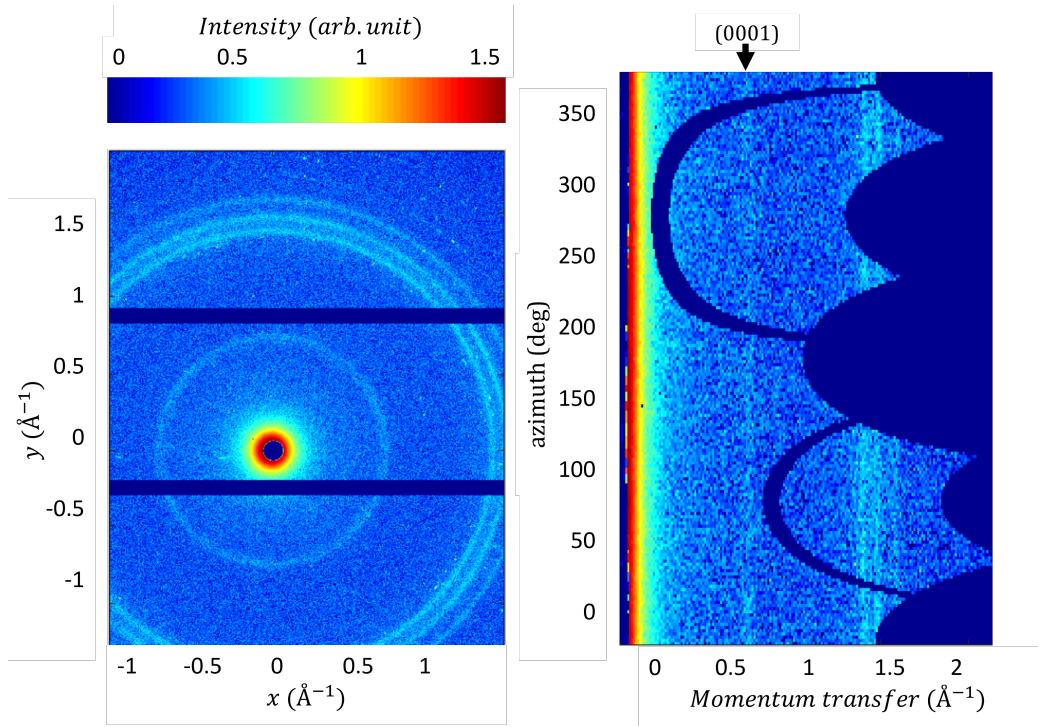


Figure 7. Radially integrated WAXS spectrum for Al 7075 after age hardening compared to calculated positions for crystal planes of MgZn_2 .

WAXS and SAXS intensity profiles before radial integration for the Al 7075 sample is shown in Figure 8. There is no major dependence of transmission based on the orientation of the sample. However, a slight variation of intensity could be identified in the WAXS measurement, see Figure 8b, mostly as a variation of intensity in the line corresponding to the (0001)-plane. This indicates a very slight preference in grain orientation for the precipitated particles. This may be a random fluctuation or a consequence of how the sample was produced.



(a) SAXS



(b) WAXS

Figure 8. Comparison between SAXS and WAXS measurements of angular dependence of transmission for Al 7075. The SAXS measurement shows no significant angular dependence, but the WAXS measurement shows a slight variation in intensity. Primarily identified as a variation of intensity in the line around $q = 0.7 \text{\AA}^{-1}$, identified as the (0001) -plane for MgZn₂.

3.3 Tensile tests for LDPE

Figure 9 shows the tensile test for ten LDPE samples in each direction. The plastic has a higher ultimate stress in the MD direction, but is more elastic in the CD direction. Table 2 shows average tensile data for ten LDPE samples in each direction.

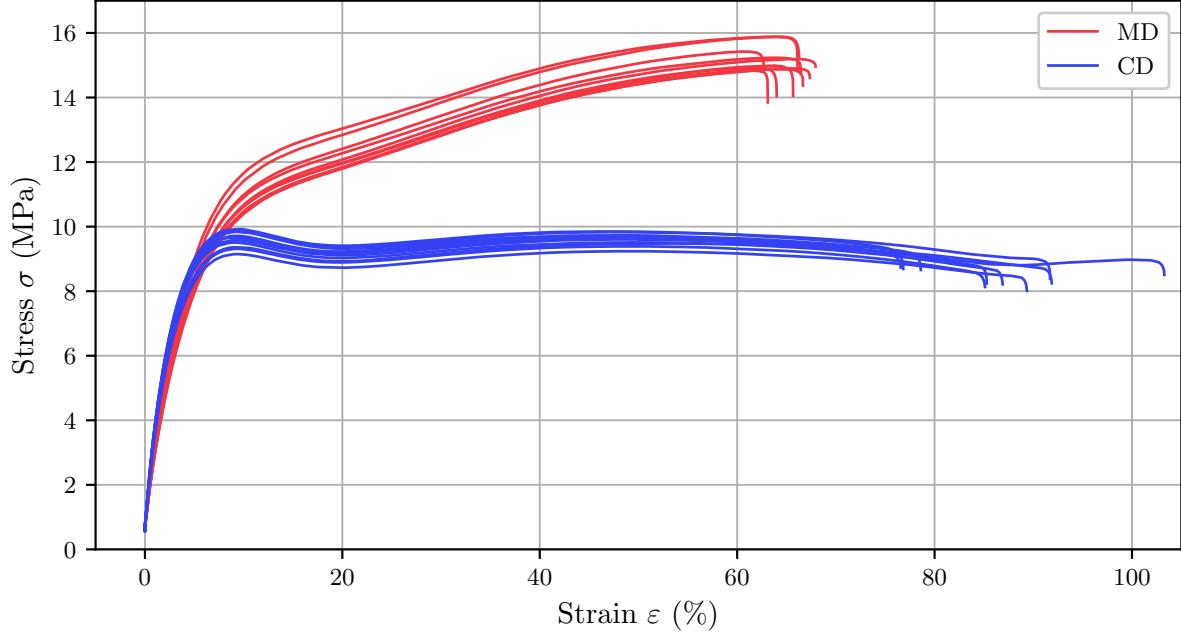


Figure 9. Data from tensile tests for the LPDE in the MD and CD directions.

Table 2. The stress $\sigma_{0.2}$ at the point of 0.2 % elongation (proof strength), ultimate tensile strength σ_{UTS} , and the strain ε_f at fracture for the LDPE samples.

Polymer direction	$\sigma_{0.2}$ (MPa)	σ_{UTS} (MPa)	ε_f (%)
MD	1.2	15.2	65.6
CD	1.4	9.6	86.6

3.4 SAXS and WAXS measurements for LDPE

Figure 10 shows the radially integrated SAXS spectra for CD and MD polymer at the deformed and undeformed sites. All spectra show a peak around 8 nm to 10 nm believed to correspond to the thickness of crystalline lamella. For both directions, deforming the sample reduced the amplitude of the peak and broadened it slightly, also seen as a change in intensity clusters in Figure 13d. Deformation also moved peak position in the direction corresponding to smaller size, especially in the CD.

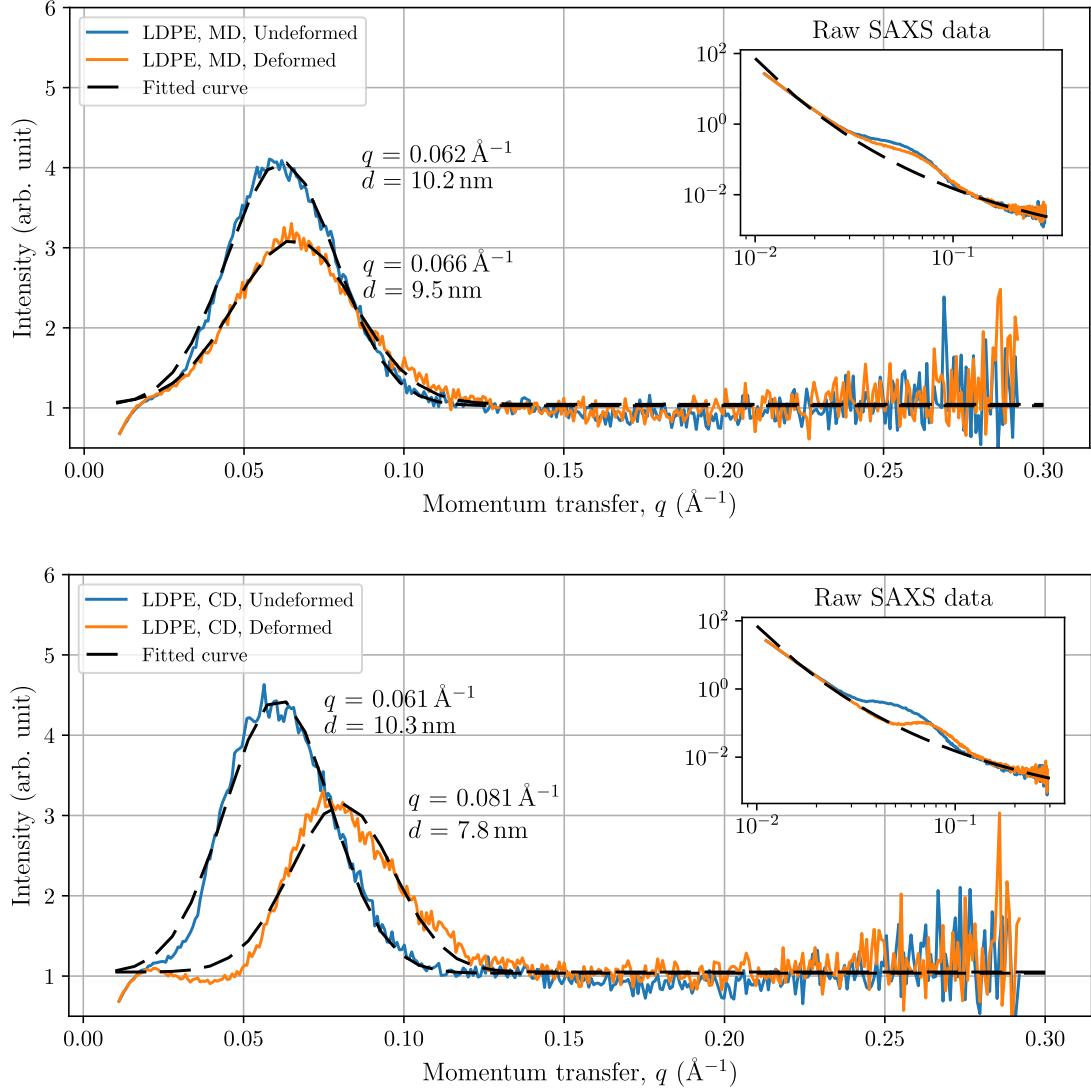


Figure 10. SAXS for the LDPE in the MD and the CD before and after tensile test. The value d differs between the peaks, meaning that the typical size of the structure in the plastic changed during the tensile test. This is most pronounced for the CD.

Figure 11 shows the radially integrated WAXS spectra for CD and MD polymer at the deformed and undeformed sites, together with expected peak positions for polyethylene crystal planes as well as TiO_2 , a common white pigment. We are not certain that the sample contains TiO_2 , but the rightmost peak could not be explained by any known crystal plane in the plastic. Deformation of the sample in the CD direction significantly reduced the amplitude and broadened the LDPE peaks. The broadening is also clear from the non-radially integrated data for CD Deformed in Figure 12. There appears to have been a shift of all peaks in MD, although we suspect that this is a calibration error since the pigment should not deform significantly.

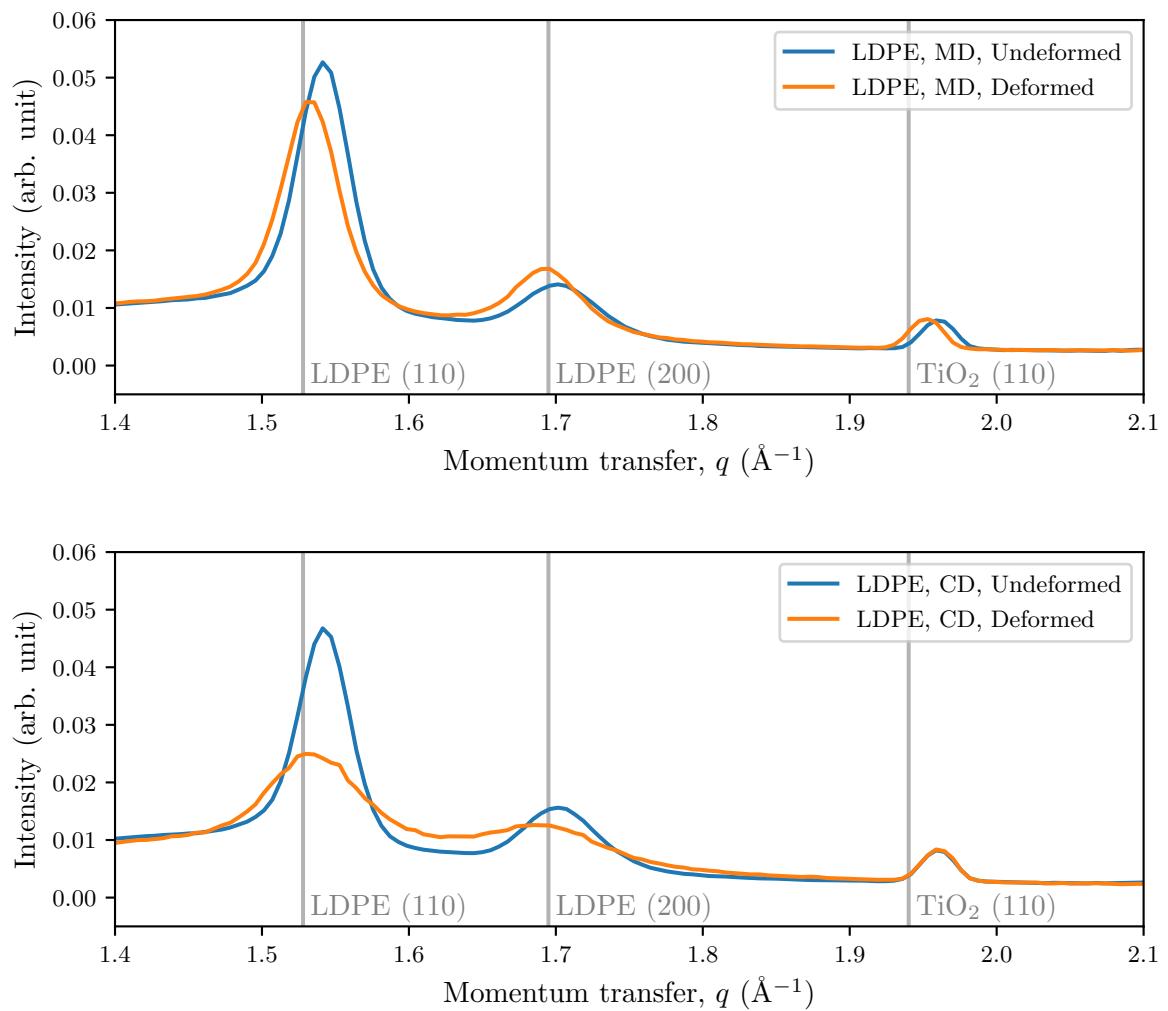


Figure 11. WAXS for LDPE in the MD and CD before and after the tensile test.

Figure 12 shows the WAXS intensity distribution before radial integration for the LDPE polymer. The deformation of the polymer increases the angular dependence of deformations, particularly in the CD. The angular dependence is larger in the CD compared to the MD for both the undeformed and the deformed polymer. It is clear that deformation of the sample makes polymers in the crystalline phase align along the deformation direction. There is also some alignment to the flow direction before deformation.

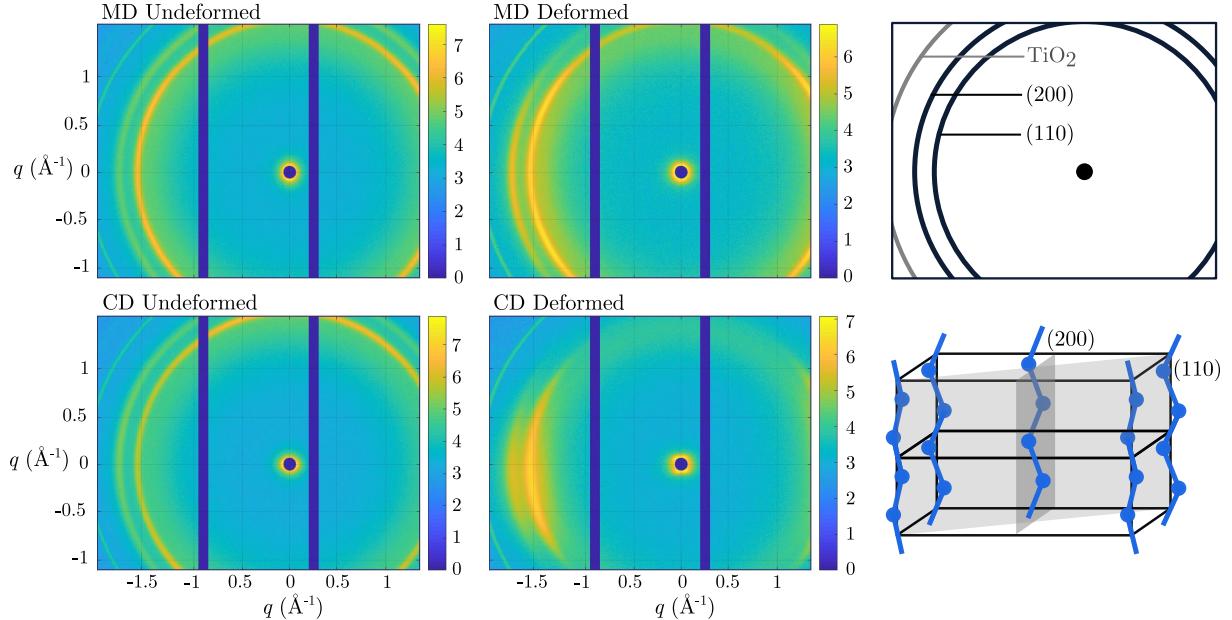


Figure 12. WAXS orientation comparison of LDPE polymer where top figures show the MD and lower shows the CD. Right top picture identifies the planes believed to be identified in the WAXS measurements. Right bottom picture shows two unit cells of the crystalline phase to visualize the orientation of the observed planes in relation to the polymer direction. The spectra show that polymers are weakly oriented along the flow direction (horizontal for CD, vertical for MD) when undeformed, and upon deformation become strongly oriented along the deformation direction (vertical).

The SAXS measurement shown in Figure 13 display a similar behavior as seen in the WAXS measurement. In SAXS, we see the orientation of the entire lamella rather than individual crystal planes. For the undeformed regions it is clear that the lamella are oriented perpendicular to the flow direction in both CD and MD. Comparing this to the WAXS result, we gather that individual polymers in the crystalline phase are generally aligned perpendicular to the lamella, which is expected. The deformed polymer displays a larger angular dependence in CD than MD. Specifically, the change shown in Figure 13d indicates that the orientation of lamella in the CD after deformation has become perpendicular to the deformation direction, meaning that the deformation significantly restructures the material. The change is not as drastic in MD, where the directionality is only slightly reduced after deformation.

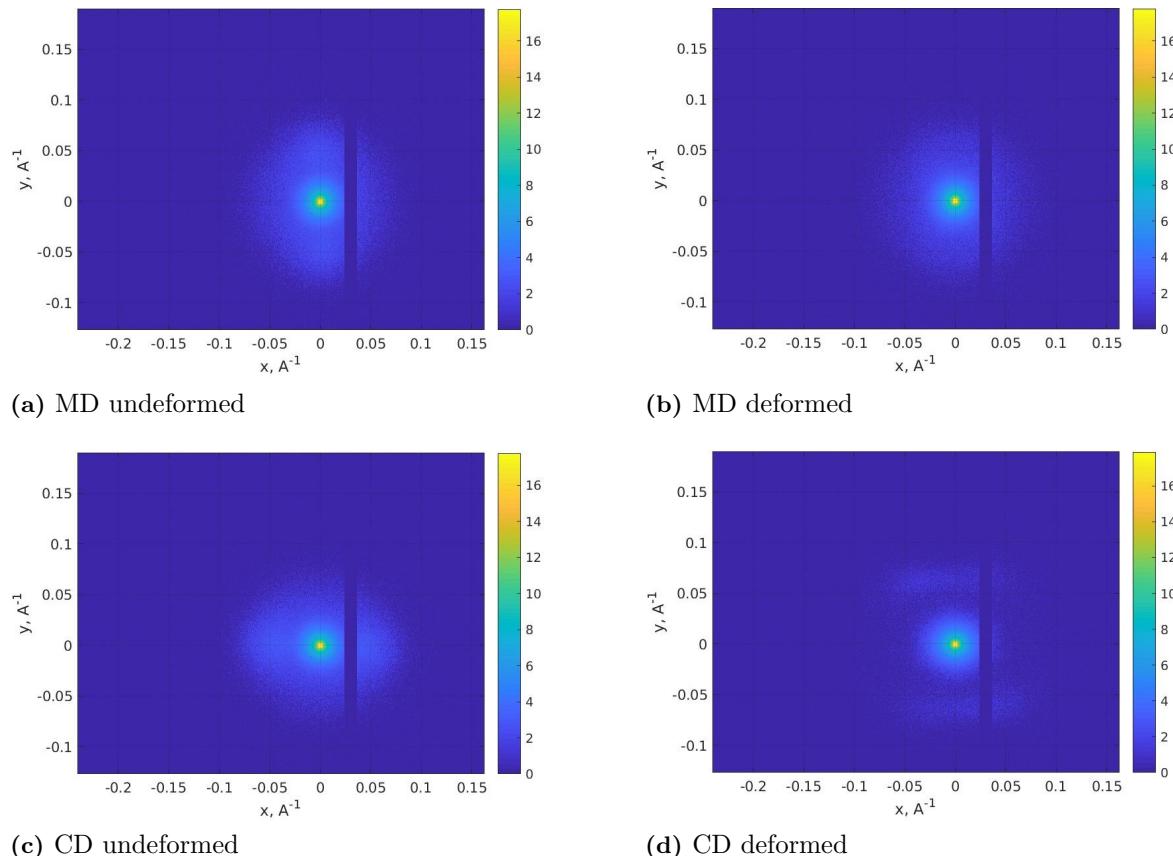


Figure 13. SAXS orientation comparison of polymer LDPE for the MD and CD, both undeformed and deformed. The difference in orientation of the oval shape in the undeformed MD and CD is interpreted as an effect of the flow direction during injection molding. After deformation, Figure 13b shows a small change in MD and Figure 13d shows that the orientation of the layers of crystalline lamella in the CD have changed significantly during the deformation.

4 Discussion

4.1 Age hardening of Al 7075

Age hardening of the Al 7075 piece resulted in higher ultimate tensile strength but lower ductility. From the WAXS measurement, it appears that precipitation of MgZn₂ occurred during the heat treatment. The precipitations hinder the movement of dislocations which means that a higher stress is required to propagate dislocations. This is why the strength of the material increased. The ductility, on the other hand, decreased since the stress needed to propagate the dislocations may become greater than the stress needed to separate atomic bonds in the material. This leads to a smaller strain at fracture for the age hardened alloy compared to before heat treatment. All in all, age hardening made the Al 7075 alloy stronger at the cost of lower ductility due to the precipitation of MgZn₂ which hinder the propagation of dislocations.

The discrepancy between the measured and calculated ultimate stress can be due to a number of reasons. Firstly, the values of M , γ , and α in equations (1), (2), and (3) are general values which might not be the best suited for this specific alloy. Secondly, f was very roughly estimated from the alloy composition, but the actual volume fraction of precipitates is unknown. Moreover, the theoretical model assumes a uniform grain distribution, whereas the WAXS measurement of the undeformed aluminum shows a slight preference in grain orientation for the (0001)-plane. Also, the volume fraction was approximated as the weight fraction, which is another source of error. Thirdly, the measured values lack uncertainties, and since only one sample was measured there is no proof of reproducibility in the age hardening. To more confidently compare theory with experiment, uncertainties should be known and the values should be given as averages over several tensile tests of the same alloy.

Another consideration is that the increase in strength $\Delta\sigma$ from dislocations bowing around and shearing through particles, (1) and (2), are mainly for smaller and larger particles respectively. We saw that (1) overestimated the measured $\Delta\sigma$ by a factor of about 100, while (2) underestimated it by the same factor. It is curious that neither of those two equations provide a reasonable value since the precipitated particles should be either small or large. This could mean that the theoretical models are not suitable in this case, or that the change in strength comes from other mechanisms. For example, the increase in strength might be smaller than expected due to coarsening as the original alloy grains grow in size during the heat treatment. Lastly, it is notable that (3) gives a more reasonable estimate of $\Delta\sigma$ compared to the measured, but the estimate is still almost three times larger.

4.2 Deformation of the LDPE

In MD, spherulites and shish-kebab structures are initially oriented along the deformation direction. We therefore do not expect the deformation to restructure the material significantly. This is consistent with the directionality of the SAXS spectrum, where we see signatures of lamella oriented perpendicular to the deformation direction both before and after deformation. For the radially integrated SAXS spectrum, there is only a slight shift of the peak towards smaller structure size after deformation. This suggests that the deformation does not significantly change the size of, or distance between, lamella. There is also relatively little change in the WAXS spectrum, which means that the crystalline structures remained intact. We therefore think that the main deformation mechanism in MD is either on a larger length scale, for example cross-linked and tangled polymer chains structures becoming more aligned, or on a smaller scale, with individual polymers breaking. As more and more structures become maximally aligned, an increasing number of links have to be broken to deform further, which would explain the increase in stress at large strains.

In CD, spherulites and shish-kebab structures should initially be oriented perpendicular to the deformation direction, with lamella perpendicular to it. This is consistent with the SAXS spectrum for the undeformed sample. After deformation, SAXS measurements show that the lamella have changed direction and the length scale is reduced. The directional change is explained by the microstructures being elongated in the direction of deformation. The decrease in length scale may be due to polymers being pulled out of the crystalline lamella and into the amorphous phase, making the lamella thinner. This is consistent with the reduced amplitude and broadening of crystalline peaks in the WAXS spectrum. Just as a perfectly ordered crystal in theory should produce an infinitely small peak in the radially integrated spectrum, a disordered and more randomly structured specimen is expected to yield a broader peak. Given a constant free energy difference between the amorphous and crystalline phase, this mechanism would require a constant amount of energy per unit length of elongation. This would explain the nearly constant stress as a function of strain in the tensile test.

The different behavior for the LDPE in the MD and CD in the tensile tests could alternatively be explained in the following way. Initially, the cross-linked polymer chains disentangle and at this stage the MD and CD will respond similarly. Once the yield strength is reached, the polymers are mostly disentangled and alignment becomes more important. Since the shish-kebab structure form in thread-like structures in the direction of flow, stress applied to the MD will generally be in the direction of alignment. This should lead to a higher yield stress, as strong covalent bonds will contribute to the polymer's ability to absorb the applied stress. If the stress is instead applied perpendicular to the alignment, as in the CD, van der Waals interactions between the polymer chains (that are weaker than covalent bonds) will determine the mechanical behavior and thus the yield stress decreases after the yield point.

References

- [1] C. Nordling and J. Österman, *Physics Handbook for Science and Engineering*, 9th ed. Studentlitteratur AB, 2020, ISBN: 978-91-44-12806-1.
- [2] A. S. M. Inc., *Aluminum 7075*, <https://asm.matweb.com/search/SpecificMaterial.asp?bassnum=MA7075T6>, (accessed: 2023-02-21).
- [3] P. Villars and K. Cenzual, Eds., *Mgzn2 crystal structure: Datasheet from “pauling file multinarie edition – 2022” in springermaterials*, accessed 2023-03-07. [Online]. Available: https://materials.springer.com/isp/crystallographic/docs/sd_1804282.

A Lab notes on the heat treatment of aluminum 7075

Below are the notes during the heat treatment of the aluminum 7075 pieces before the lab session at Chalmers material analysis laboratory.

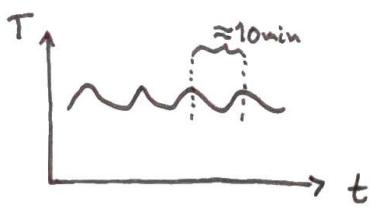
TIF326 Physics of materials

Götzfrid Olsson

Heat treatment of 7075 aluminum alloy before lab.

Goal: treat a piece  and  in an oven at 160°C for 2 hours.

- 16:44 Started the oven. Letting it come up to temperature.
Trying to hit the right $T=160^{\circ}\text{C}$. oven "display" is not very accurate.
- 17:03 Put temp at 150°C on oven. After $\sim 5\text{ min}$ it went up to 185°C on the external thermometer.
- 17:10 Put oven at 125°C . Waiting and watching temp on thermometer.
- 17:13 Opened oven door to drop temp.
- 17:14 Closed door. @ $\sim 162^{\circ}\text{C}$ on thermometer.
- 17:18 Seems more stable $\sim 160^{\circ}\text{C}$, but decreasing.
Letting it reach equilibrium and waiting more
- 17:30 Might be stable around 160°C . Now, $* < 160^{\circ}\text{C} \dots$
- 17:34 Raised oven a little bit.
- 17:47 Raised oven temp a little again.
- 18:11 Restarted the thermostat graph, put the metals into the oven (middle of). start: 18:12.
- 19:15 Temp suddenly went up a bit - though nothing was touched or changed on the oven. One more hour, then it is done.
See that oven turns on/off from the thermometer:



2023-02-02

Gottfrid Olsson

20:11 2h have passed. Taking out and quenching the metal in room-
temperate water. Finish time: 20:12

20:20 All seem to have gone well \approx 1.
Thermometer graph have been screenchoted.

B Lab notes on the SAXS and WAXS measurements

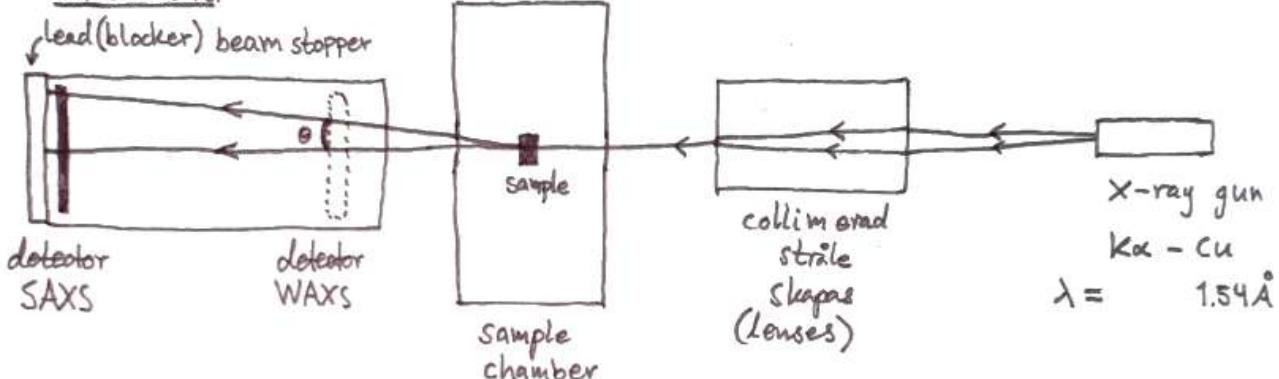
Below are the notes during SAXS and WAXS measurements of the heat treated aluminum 7075 piece at Chalmers material analysis laboratory.

SAXS and WAXS measurements lab Gottfrid Olson

10:05 Will measure on high-density polyethylene (HDPE) plastic used in Tetrapack, and on our 7075 Al sample.

Mattias Thuvander has grinder our sample so it is now very thin. (See PP on lab for details about things).

HDPE samples before and after tensile test.

Instrument:

10:20 Mounting our sample. Vacuum: < 2 mbar.

10:35 1.3 mbar. Locating our sample and reference.

LAB6 Lanthium hexaboride is the reference sample.

"G2-LAB6-WAXS-1min" filename. Measured at <1.2 mbar.

Dark bands in measured data because detector is made of three smaller detectors with gaps between them. Can fill in those gaps by symmetry argument.

10:50 Locating our sample. Measuring at the edge, ≤ 1.12 mbar

"G2-AL7075-WAXS-5min".

11:01 Check raw data for orientation in sample.

Then radially integrated data. What does the peaks correspond to in the structure of the material? Peak position $\rightarrow q \rightarrow d = \frac{2\pi}{q}$.

11:05 Peak width: strain, more grain boundaries. A perfect crystal would create δ -function peak; random polymer would result in a wider peak.

changing to SAXS.

11:10 "G2-AL7075-SAXS-30min", ≤ 1.11 mbar.

Break.

11:40 Measurement done.



Radially integrated data: Look for change in slope. What structure in material can have such size?

11:45 Background is linear in log-log. Data - background fit = gaussian peak we are looking for

Focus on qualitative analysis "this larger than that" rather than specific values.