ME-865 : Quantification of martensite fraction in high strength steels by X-ray diffraction

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Aims of the experiment and scientific background

The need for making lighter structures in the automotive industry for lowering the fuel consumption requires the development of new generations of steels offering both a very high strength and a good formability. Such steels are being currently developed based on the Fe-Mn-C system, which satisfy both criteria.
The stable crystallographic structure at high temperature of these steels is γ austenite (FCC). Upon quenching to room temperature, a number of phases can be present (fig. 1):

- γ austenite, metastable
- ε martensite, a hexagonal metastable phase
- α' martensite, a tetragonal body centered phase

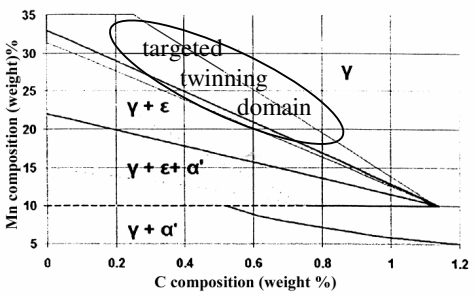


Figure 1 : Structural state of the steel after deformation at room temperature, as a function of Mn and C composition.

The transformation of γ to either α' or ε is a diffusionless displacive transformation. The product is extremely hard and brittle. The properties of these steels are critically dependent on the deformation mode of the austenite :

- deformation by dislocations slip ;
- deformation by twinning (controlled by the stacking fault energy, thus by the composition) [1] ;
- deformation by transformation into martensite (stress induced transformation or TRIP effect) [2].

The best combination of properties is obtained by twinning. However, this deformation mode is very close in terms of processing to the domain of martensitic transformation, which results in the steel embrittlement.

The quantitative characterisation of the proportion of the different phases as a function of composition and amount of deformation is not an easy task : when present, martensite is extremely fine (laths less than 100 nm in thickness), and both ε and α' may be present simultaneously. X-ray diffraction is particularly adapted to this problem [3].

Experimental

Five series of different steels have been investigated (all compositions in wt%) :

- A : 17%Mn-0.4%C with three different levels of deformation by rolling. A small amount of martensite is expected to be present in the quenched state and a large amount of ε and α' in the deformed state.

- B : 17%Mn-0.95%C . It is not expected to contain any martensite in the quenched state but ε in the deformed state.

- C : 17%Mn-0.95%C + Si (from 0% to 1 % Si). Influence of Si composition has been investigated.

- D : 22%Mn-0.4%C (all in weight %). It is also expected to contain no martensite in the quenched state but ε in the deformed state.

- E :The same as above, but coming from deep drawn parts and thus highly deformed

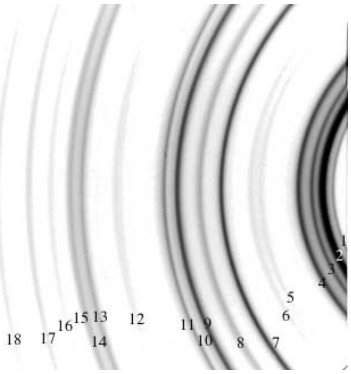
All samples have been characterized in transmission with an X-ray energy of 20 keV (ensuring a penetration depth of about 50 μm) and with a 2D detector. Two distances of the detector have been used: 9 cm (low resolution) and 50 cm (high resolution). Three samples have been analysed with a point detector placed behind shits (horizontal: 16 mm, vertical: 0.15 mm) too in order to calibrate the 2D detector. High resolution analysis has been performed with a spinning of the sample, limiting thus texture effects and improving the diffracting volume. Texture of samples has been studied from low resolution analysis with no spinning of the samples. The use of the 2D detector has allowed a high gain of time analysis: for one

sample, a high resolution analysis is performed only in 1 h 20 with the 2D detector whereas it lasts more than 10 h with the point detector.
A computer software (DAD) [4] has been modified with the help of D2AM staff to transform 2D data in a 1D spectrum, including correction due the variation of transmission of the samples with the angle of diffraction.
Some problems have been encountered with spatial correction of the 2D detector. So a new grid has been measured and the detector has been recalibrated. Even if the resolution of the detector has been improved, this new calibration does not still give entire satisfaction, and studies are under way to improve it.

Results

Determination of phases proportion :

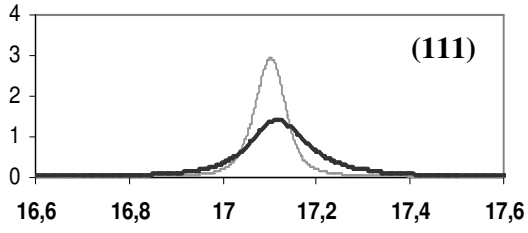
This has been achieved using the ratio of intensities of different peaks. Martensite have been found in steels of the series A: less than1 % of ε and no α' in the as quenched steel, 2 % of ε and little traces of α' after 6.5 % of deformation and 11 % of ε and 2 % α' after 32 % of deformation. In all other series, no traces of martensite have been found, even in the most deformed samples. This indicates that compositions of those steels are in the good range to avoid martensitic transformation.

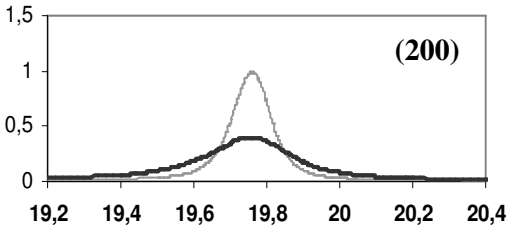


2d spectrum of samples from series A with 32 % of deformation where the three phases are present

Peak profiles

Even if the correction of spatial distortion is not complete yet, semi-quantitative results can be obtained. For all series, we notice, as expected, that the width of austenite peak increases with deformation. But this broadening is not homogeneous: γ (200) becomes much larger than γ (111). Moreover, we observe also an inhomegeneous shifting of the Bragg angle of only some of the γ peaks. Such a modification of the diffraction profile is typical to the formation of a high density of defaults such as stacking faults, twins and partial dislocations. These results are promising in the aim of determining densities of crystalline defects.





Spectrum (point detector) of slightly deformed (gray) and highly deformed (bold) of samples of series D and E. We can notice a shift of the (111) γ peak (left) whereas the (200) γ peak(right) remains at the same position

Conclusion

These experiments have allowed to improve the procedure of powder diffraction analysis at D2AM, both for the 2D detector calibration and for the treatment of 2D data. Important results on the presence of martensite depending on the steels compositions have been obtained. We can hope from first treatment of peak profiles that others results will come on the properties of defaults in the microstructure.

References

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