Correlation of Material Phase Hardness and Macroscopic Properties of 1180DP Steel

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The microhardness and single grain hardness of two heat treated dual-phase steel products were examined. Two methods of measuring individual phase properties were utilized; performing a grid of indentations and targeting the hardness of indentations made in isolated phases. Nanoindentation data indicated hardness in heat treatment one of 3.9 GPa for ferrite and 6.2 GPa for the hard phase. Material receiving heat treatment two contained ferrite measuring 3.9 GPa and the hard phase at 6.1 GPa. Differences in yield strength, tensile strength and hole expansion ratio were not found to be associated with differences in phase hardness.

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Project Background

Advanced High Strength Steels (AHSS) combine high yield and tensile strength with good formability. ArcelorMittal's 1180DP product is a dual-phase product and it is offered as galvanized and uncoated product with two different heat treatments. The compositions and properties are:

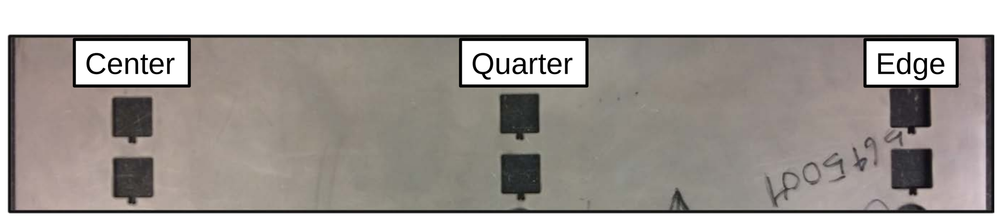
	C	Mn	Si	Other	YS (MPa)	TS (MPa)	HER
HT1	0.13	2.5	0.7	Confidential	850	1240	23.8%
HT2	0.14	2.1	0.7	Confidential	910	1250	29.5%

ratio (HER) of galvanized and uncoated 1180DP steel

Forming operations risk the creation of cracks and early material failure problems. Deformation behavior of dual-phase steels can be attributed to the yielding of ferrite and fracture of harder phases. Phases include martensite and bainite, depending on the heat treatment. Achieving a high HER requires a limit in the variation of phase hardness. For these products, bulk and nanohardness data was collected and SEM analysis was performed to gain knowledge in the contribution of each phase to macroscopic behavior.

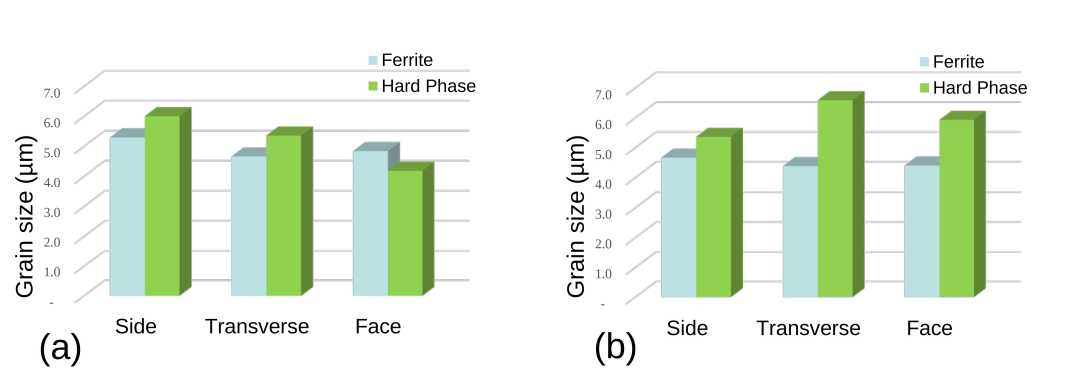
Experimental Procedure

Samples from different locations on the sheet width were cut via a water jet. They were mounted and polished to a 3 micron diamond stage and etched with 0.5% and 5% Nital (nitric acid and ethanol).



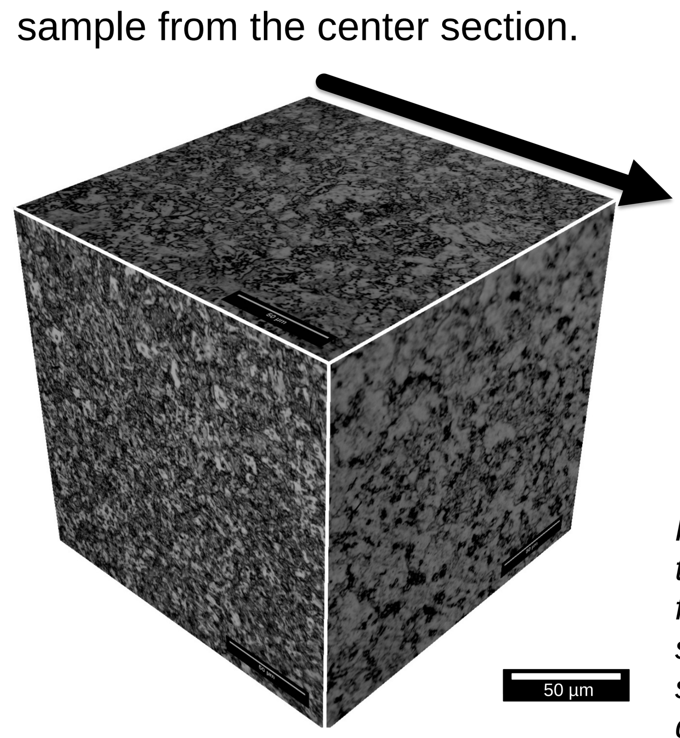
Samples acquired from three positions on a half sheet of rolled steel. A directional tab on removed samples documents the rolling direction.

One sample set was etched and examined via optical microscopy to analyze grain size and phase fraction for each product and location. The Heyn Intercept Method was used to measure grain size. 48% of the volume fraction (VF) of HT1 was ferrite and 52% of the VF of HT2 was ferrite.



HT1 (a) and HT2 (b) grain sizes from quarter section. Grains were not determined to be shallow compared to their observed surface area. 95% confidence intervals ranged from 0.76 μm to 1.16 μm for ferrite and 1.18 μm to 1.82 μm for harder phases.

The microstructure of HT1 material is shown, based on a sample from the center section.

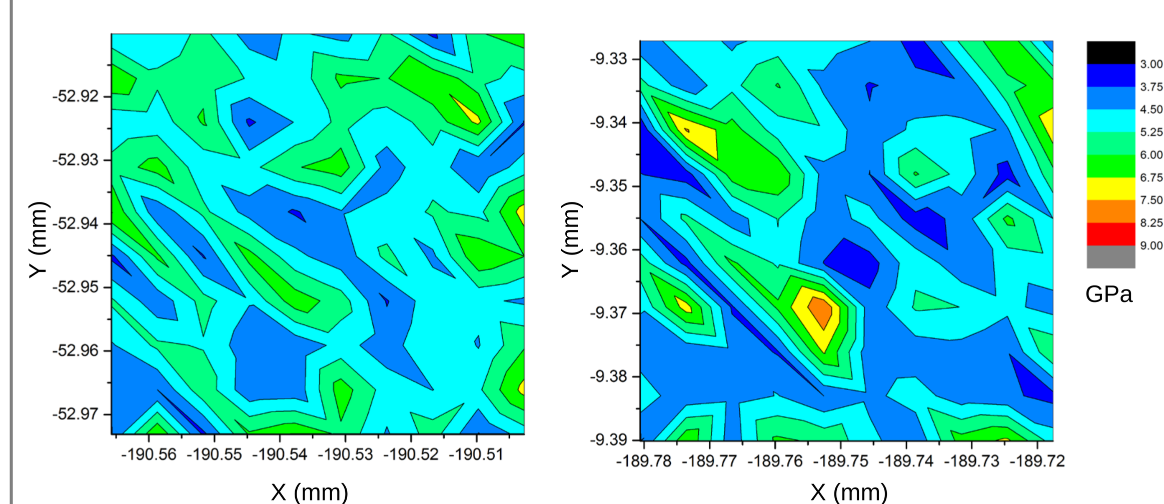


Rolling direction indicated with the arrow. Scale bars on each face represent 50 μm. Grains showed minor variability in shape and orientation on different faces.

A second set of samples were prepared for nanoindentation with a Berkovich-tipped probe. These were etched using 5% Nital for 1.5 seconds. Two methods were used to measure the hardness of grains: random grid sampling with post-indentation phase analysis and direct targeting of isolated phases. After nanoindentation, a diamond scribe was used to mark grid locations to aid in finding them for SEM imaging.

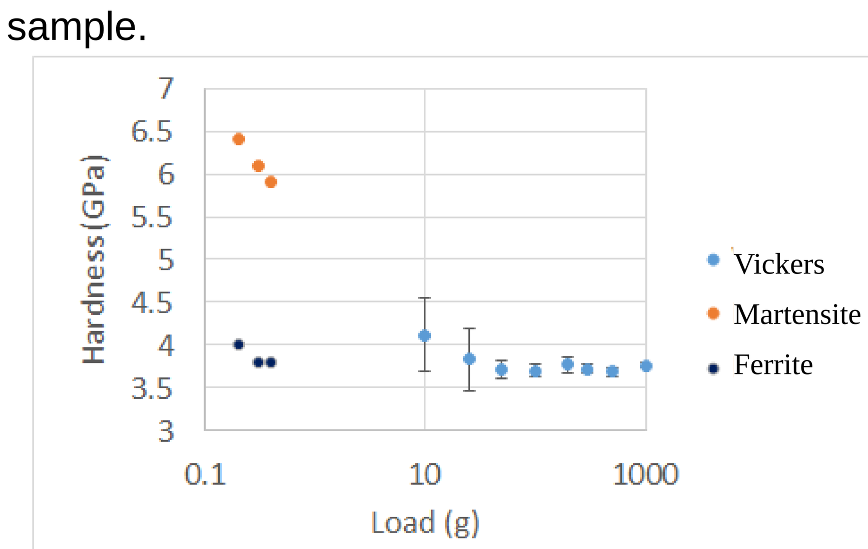
Results

Nanoindentation tests utilizing a 10 by 10 grid of test points yielded the following results.



Hardness maps of HT2 (L) and HT1 (R) material, quarter section; data recorded with 3000 μm load and third loading segment. In both cases product results in islands of the hard phase separated by a sea of ferrite due to diffusivity time.

The nanohardness of ferrite and the hard phase displayed a similar trend to the measured Vickers hardness of a ferritic steel sample.



As the applied load of Vickers hardness increased, readings approached the measured hardness of ferrite obtained from nanoindentation. Vickers loads of 10g have high variation, suggesting the measurement of multiple phases. Data taken from uncoated edge sample.

The following hardness data was obtained for each method using loads ranging from 2.350 to 2.450 mN.

Grid Method	Ferrite (GPa)	St. Dev.	95% CI	Hard phase (GPa)	St. Dev.	95% CI
HT1	3.95	0.29	0.07	6.37	0.79	0.13
HT2	4.05	0.43	0.10	6.47	0.72	0.14

IPT	Ferrite (GPa)	St. Dev.	95% CI	Hard phase (GPa)	St. Dev.	95% CI
HT1	3.97	0.42	0.36	5.31	0.14	0.14

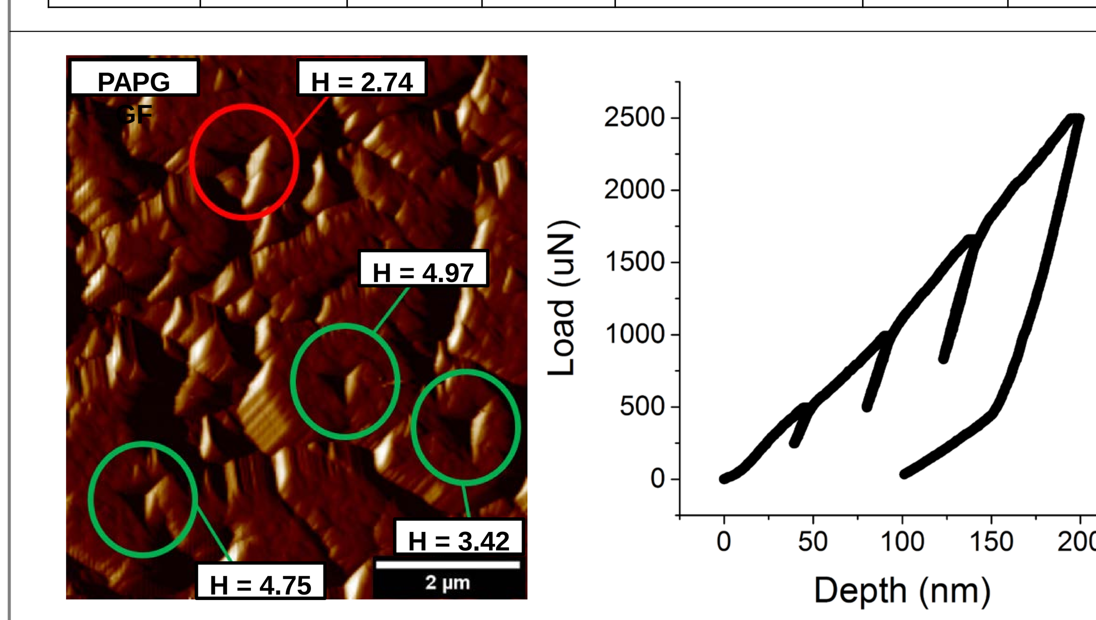
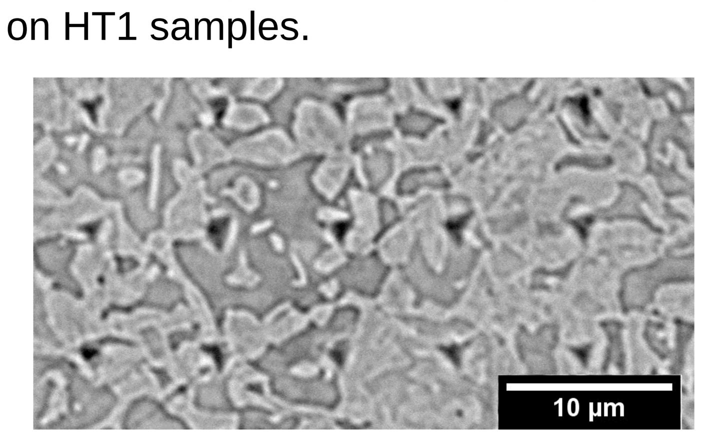


Image from the scanning probe on the Hysitron. The lower three samples, highlighted in green, are indicative of usable placements on ferrite. The indentation at the top was placed too close to a grain boundary. A sample load-depth curve is shown to the right.

Isolated phase targeting was completed with HT1 using a load of 1000 μN. To maximize the amount of data obtained for one material, all isolated phase targeting experimentation was completed on HT1 samples.

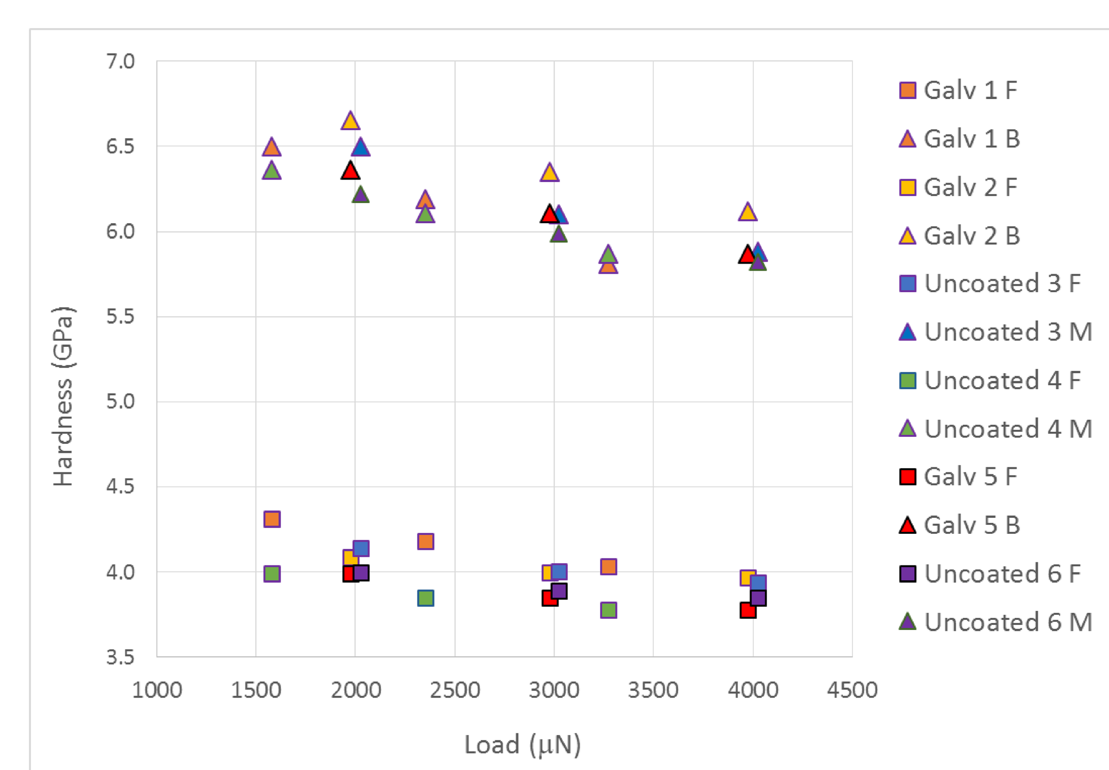


In this SEM image, ferrite can be seen as the darker phase and the lighter phase represents the harder phase. Indents can be observed within grain boundaries.

Discussion

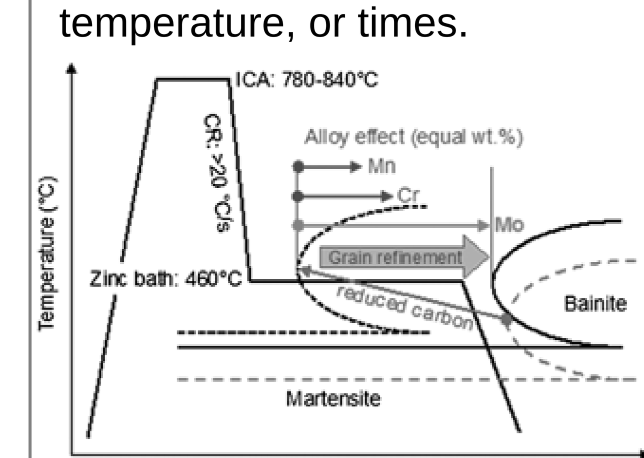
Nanoindentation tests with multiple loads indicated that a size effect problem exists on initial loading of the material. The lack of a fully developed plastic zone results in abnormally high hardness for the first loadings. Hardness was best obtained on the third or fourth loading of the material, particularly when these loads occurred between 1500 and 2500 μm.

Isolated phase targeting on etched samples yielded a small number of usable indentations. These were used to confirm ranges identified with the grid sampling method, which produced a higher yield of usable indentations per hour of machine time. Key factors affecting the quality of nanoindentation results included the size of the grains relative to the size of the indentation probe, the roughness of the sample surface after polishing, and the extent to which grains were level with the surface.



Displayed hardness values for hard and soft phases are based on average measurements from accepted indentations from the grid sampling method.

No statistically significant difference was observed between the hardness of either hard phase in the two products, although measurements from hard phases from HT1 were slightly elevated. Phase hardness differed by 2.25 GPa in HT1 and 2.20 GPa in HT2. These variations are likely due to variations in the microstructure formation during the heat treatment process. HT2 has a HER of 29.5% compared to 23.8% for HT1. Cracks during hole expansion operations typically form at the interface between hard and soft phases. Individual peak hardness in grains affects HER more than average hardness. The presence of a low number of harder grains in HT1 may be increasing the likelihood of crack formation and growth. It may be possible to increase the HER for HT1 by adjusting the process chemistry, temperature, or times.



Alloying effects on bainite formation in low-carbon DP steel during hot dip galvanizing cycle. Microalloyed Low-Carbon Multiphase Steels, DOI: 10.1002/srin.201500352

Recommendations

The grid method of sampling is recommended for nanoindentation, combined with SEM imaging for phase analysis. Grid spacing should exceed the average grain size. The grain volume sampled by the indenter should be less than 15% and indentation data should be excluded when the observed grain diameter is less than 50% of known grain size. Sample preparation is critical for the collection of high quality results.

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