NANOINDENTATION MEASUREMENTS OF THE INTERMETALLIC PHASES IN GALVANNEAL COATINGS

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1. Introduction

Galvannealed IF-HS steel sheets are presently the subject of interest in automotive as well as steel industry for higher corrosion resistance, and considerably higher strength with excellent formability[1]. These coatings may contain one or more of four possible iron-zinc phases of low thickness[2]. The mechanical properties of these phases haven't been in detail investigated. Thus the depth sensing indentation is a suitable method for measurements of mechanical properties of these phases.

2. Experimental details

IF steel (galvanized) was used for the experiment. One sample was prepared from this galvanized steel and next the steel was additionally annealed in the furnace at 450 and 500 °C at 10 and 60 s holding times, respectively. The steel thickness was 0,85 mm and average thickness of the zinc layer was 13,5 μm. We marked these samples as R456 and R501. Then cross-sectional samples were prepared via classical metallographic method. All samples were investigated via scanning electron microscopy (SEM) and energy dispersive X-ray microanalysis (EDX) to determine the composition of individual intermetallics[3].

Nano Indenter XP with Berkovich indenter was used for the hardness and indentation modulus measurement. Load control mode was chosen and the load of 10 mN was used. Firstly the galvanized sample was measured and the mechanical properties of both, steel substrate and zinc layer were evaluated. Then two galvannealed samples were measured. Twenty indentations in each intermetallic phase were performed on the samples. In the case of thin Γ layer the load of 5 mN was used because of achieving the appropriate results from smaller indents.

3. Results and discussion

Firstly the mechanical properties namely the hardness and indentation modulus of galvanized steel were measured. Twenty indentations in both, steel and zinc layer (η-phase) have been performed. The load of 10 mN was used. The measured values of the indentation hardness H_{IT} and the elastic modulus E_{IT} are in the Tab. I.

Then two galvannealed samples were measured using load control mode. Approximately twenty indentations into each intermetallic layer were performed using the load of 10 mN.

Table I
Hardness and modulus values of galvanized steel and zinc layer

R – zakl	Average hardness [GPa]	Average modulus [GPa]
Steel substrate	2,13	200
Zinc layer	0,72	102

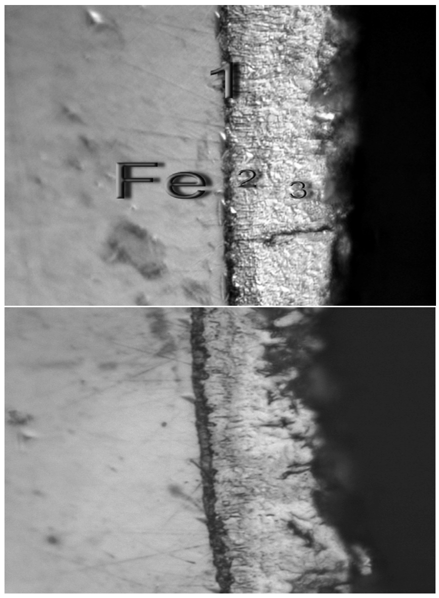


Fig. 1. **Steel with visible zinc layer - illustration of marking of measured intermetallic layers**

We named the intermetallic phases using the numbers instead of Greek alphabet, Fe – steel substrate, 1 – Γ layer, 2 – Γ+δ layer, 3 – δ layer see Fig. 1 and Table II. There is a great difference between hardness of intermetallic layers of galvannealed steel when compare with zinc layer of galvanized steel.

Table II
Hardness and modulus values of individual intermetallic phases for each measured sample

	R - zakl		R 456				R 501			
	steel	zinc layer	steel	1	2	3	steel	1	2	3
H_{IT} [GPa]	2,13	0,72	2,44	1,36	4,58	3,33	2,39	1,11	5,12	4,46
E_{IT} [GPa]	199,8	102,1	204,3	129,3	159,0	116,9	215,2	106,3	138,4	120,9

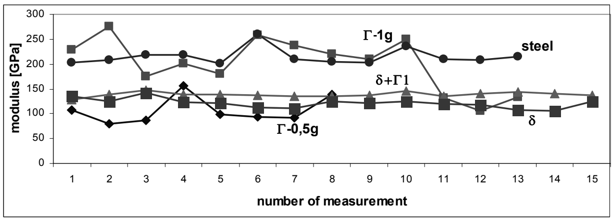


Fig. 2. **Elastic modulus measured by Nano Indenter XP – sample R456**

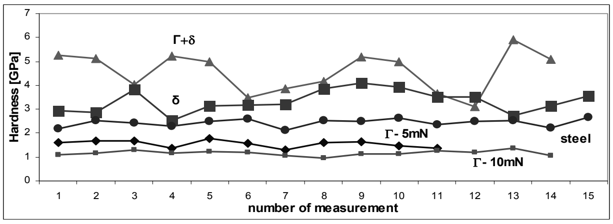


Fig. 3. **Hardness measured by Nano Indenter XP – sample R501**

Table III
The averages values of indentation hardness, comparison of literature[3] and measured values

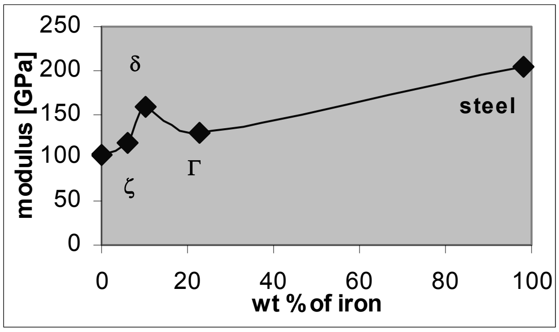
	HV	H_{IT}-calculated		E_{IT} - measured		H_{IT}- measured	
				450 °C/60 s	500 °C/10 s	450 °C/60 s	500 °C/10 s
η	52	0,56	η	102		0,72	
	39	0,42					
	71	0,77					
ζ	208	2,25	ζ				
	117	1,26					
	177	1,91					
δ1	358	3,87	δ	117	121	3,33	4,46
	273	2,95					
δ0	270	2,92	δ+Γ1	159	138	4,58	5,12
Γ1	505	5,46	Γ	129	106	1,36	1,11
Γ	326	3,52					
Fe	104	1,12	Fe	204	215	2,44	2,39

The average values of the indentation hardness and elastic modulus are in the Tab. II. The same measurement was performed for the second sample. The hardness and indentation modulus of the phases have the same trend as that for the first sample.

As you can see in Fig. 1, the indents in Γ layer are so expansive that they interfere into the base material but also into the Γ+δ phase. The thickness of Γ layer is varying from 500 to 2200 nm for used temperatures of annealing and holding times moreover this layer is not continuous and there are some discontinuities[3,4].

Thus the load of 5 mN was used for reaching the appropriate properties of this thin layer. According to literature data the hardness of the Γ layer is much greater than the hardness of the others intermetallic layers. In our experiment the values for the Γ layer are the lowest ones. But as can be seen in the Fig. 2 the values for the modulus of each layer are typical for Fe-Zn. More experiments with lower loads are needed for achieving the appropriate values of the mechanical properties of the Γ layer. It is evident that the hardness of intermetallic layers decreases with the decrease of the content of the Fe in the layer, except of the Γ layer.

When the load of 10 mN was used for the measurements of the thin Γ layer, the hardness values reach the steel hardness values. Bigger part of each indent interferes into the base material and only little area reach the layer. Also the values of the modulus are typical for the steel. But in the case of 5 mN load, the hardness and modulus values are as low as for the first sample, see Tab. II.



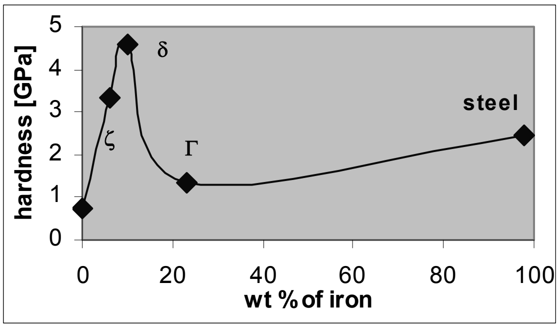


Fig. 4. **Indentation hardness and indentation modulus values in dependence of the weight percentage of the iron in zinc layers, sample 456**

When compare to the literature value for Vickers measurements using 250 mN load and conversed into H_{IT} values and H_{IT} values from our indentation measurements there are not great differences, see Tab. III.

In the Fig. 4 it can be seen that the course of indentation hardness and indentation modulus are not in accordance with the course of the weight percentage of the iron in the intermetallic layers. Such as the Γ layer possesses the highest percentage of the iron, low values of the hardness and modulus are measured in our experiment.

4. Conclusions

Depth sensing indentation is a suitable method for the detail experimental identification of local mechanical properties of intermetallic layers in zinc coating on galvannealed steel sheet.

There are not great differences in the Vickers microhardness values conversed into the indentation hardness and measured values of H_{IT} using depth sensing indentation method.

The annealing has the meaningful influence on the mechanical properties of the zinc layer which is transformed into the intermetallic layers. The formation of intermetallic layer causes the increase in hardness and elastic modulus.

The change in the used annealing temperatures and holding times does not have great influence on the mechanical properties of Intermetallic layers. Values for both samples are quite similar.

The weight percentage of the iron in the intermetallic layers is not in accordance with the indentation hardness and indentation modulus of these layers.

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Z. Vadasová[a], L. Pešek[a], M. Kollárová[b], O. Bláhová[c], and P. Zubko[a] *([a] Faculty of Metallurgy, TU of Košice,Slovakia, [b] Research and Development Centre, U. S. Steel, Košice, Slovakia, [c] West Bohemian University, Plzeň, Czech Republic):* **Nanoindentation Measurements of the Intermetallic Phases in Galvanneal Coatings**

The paper deals with indentation measurements of hardness of the intermetallic phases in the galvanneal coatings. Galvannealed IF-HS steel sheets are presently the subject of interest in automotive as well as steel industry for higher corrosion resistance, and considerably higher strength with excellent formability. These coatings may contain one or more of four possible iron-zinc phases of low thickness. The mechanical properties of these phases haven't been in detail investigated. Thus the depth sensing indentation is a suitable method for measurements of mechanical properties of these phases.