

Traceability of the amount of Phosphorus in NiP-coatings

1.

Self-supporting NiP-foils with nominal amounts of 3-12 wt% P and thicknesses ranging from 3 μm to about 25 μm were obtained from the company Atotech in Berlin. Selected areas of these foils were analyzed by XRF and preliminary XRF-values for average NiP-thickness and average percent of P were obtained.

During this step all foils were checked for homogeneity and possible gradients on front- and back side of the foils. Typically the foil sizes range from 15x15mm to 50x50 mm and the number of analyses per foil range from 25 to about 100 analyses points.

2.

3-6 Aliquots of each foil, whose weight was carefully determined, were analyzed by ICP-OES for their absolute amounts of Ni and P (given in element-wt%). Aliquots of the same foil were sent to three ISO-certified chemical laboratories and the results for wt% P agreed between labs to within 2-5% relative. Typically 2 sister-pieces of the foils sent for wet chemical analyses were kept as XRF-Master foils.

3.

Now, knowing the average amount of P for each original foil the preliminary average XRF values for P were corrected accordingly for the XRF-Masterfoils, which were not sent for ICP analyses.

4.

With the correct amount of P known for each Master-foil, the correct foil-thickness of these Master-foils was calculated from additional XRF measurements.

5.

Two Fe-sheets coated with NiP were treated in a similar way. The NiP-coating was removed from the back side and from the edges and the average P-content and NiP-thickness was determined by averaging about 150 XRF-analyses per metal sheet. Subsequently the metal sheets were cut in 4 pieces with well-known x- and y-dimensions and three (respectively 2) aliquots were sent for wet chemical analyses of Ni and P. Again, with the known amount of Ni and P the preliminary XRF values of the remaining sister pieces were corrected.

The following table gives an overview of the presently available NiP-samples (foils or sheet metals) which were independently analyzed for their Ni and P inventory.

Overview of NiP-Master-samples				
NiP "Master"-foils				
Nominal values		Code	wt% P s from ICP analyses	thickness
2µm, 12%P	part 1	ADJFZ	10.6% ± 0.5%	2.94 µm ± 0.03 µm
		ADJFY	10.6% ± 0.5%	2.89 µm ± 0.01 µm
	part 2	not mounted	10.7% ± 0.5%	2.79 µm ± 0.01 µm
5µm, 12%P	part 1	not mounted	11% ± 0.5%	5.74 µm ± 0.07 µm
	part 7	ADJGN	11.2% ± 0.6%	5.88 µm ± 0.03 µm
		ADJGM	11.2% ± 0.6%	5.75 µm ± 0.03 µm
		ADJGL	11.2% ± 0.6%	5.98 µm ± 0.05 µm
10µm, 12%P	part 7	ADJGO	11.3% ± 0.6%	11.19 µm ± 0.13 µm
		ADJGP	11.3% ± 0.6%	11.29 µm ± 0.06 µm
	part 8	not mounted	11.3% ± 0.6%	11.06 µm ± 0.05 µm
20µm, 12%P	part 3	ADJGS	10.5% ± 0.5%	27.35 µm ± 0.51 µm
	part 4	not mounted	10.6% ± 0.5%	24.81 µm ± 0.55 µm
Fischer Foil (10.2 µ, 8.5 %P		Fischer Foil Side B	8.5 % ± 0.4%	9.52 µm ± 0.09 µm
20µm, 8%P	part 2	not mounted Side B, (=V)	7.9 % ± 0.4%	22.62 µm ± 0.38 µm
20µm, 4%P	part 2	not mounted Side V	2.5% ± 0.2%	16.76 µm ± 0.25 µm
		part 3	not mounted Side V	2.5% ± 0.2%
10µm, 4%P	part 2	not mounted Side V	2.5% ± 0.2%	7.04 µm ± 0.06 µm
Foil CNN, 20µm, 8%P	part 3	nicht montiert Side A	7.44% ± 0.2%	25.2 µm ± 0.9 µm
20µ, 4%, EMPA (electron microprobe analyses)		Seite R	2.51%	18.1 µm
		Seite V	3.63%	
NiP/Fe "Master"-sheet metals				
Nominal values		Code	wt% P	thickness
20µm, 8%P	part D	20µm, 8%P	8.9% ± 0.5%	31.75 µm ± 0.5 µm
5µm, 4%P	part C	5µm, 4%P	2.2% ± 0.1%	5.92 µm ± 0.03 µm