FMR study of influence of nitriding process of promoted nanocrystalline iron and nitrides reduction process at various nitriding potentials

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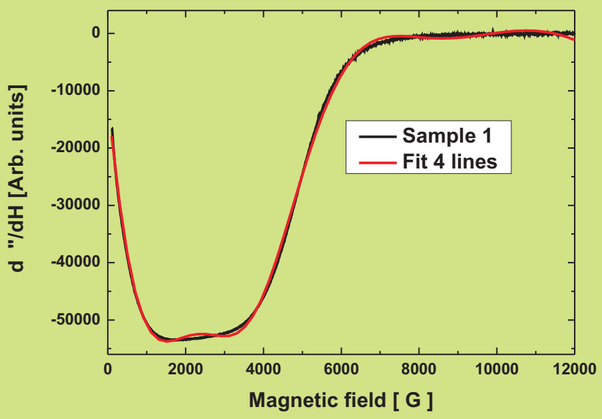
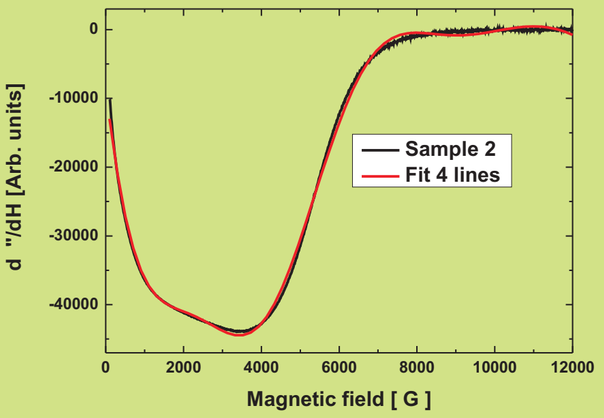
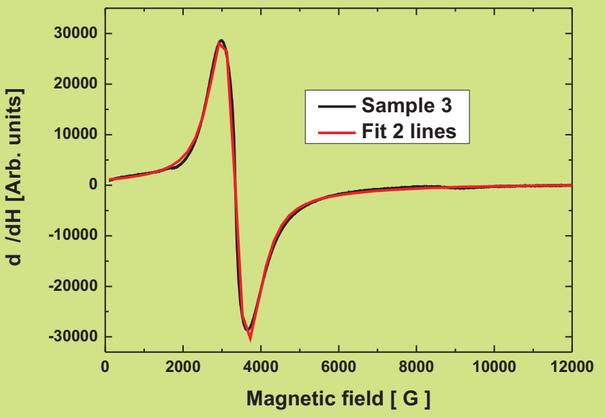
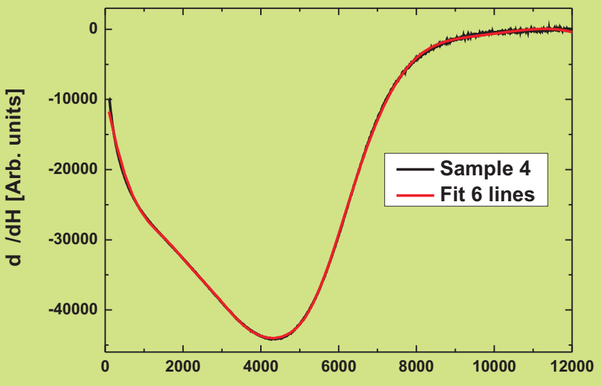
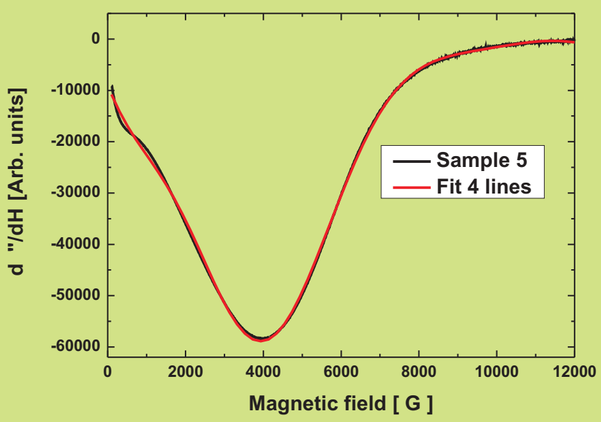
FMR spectra

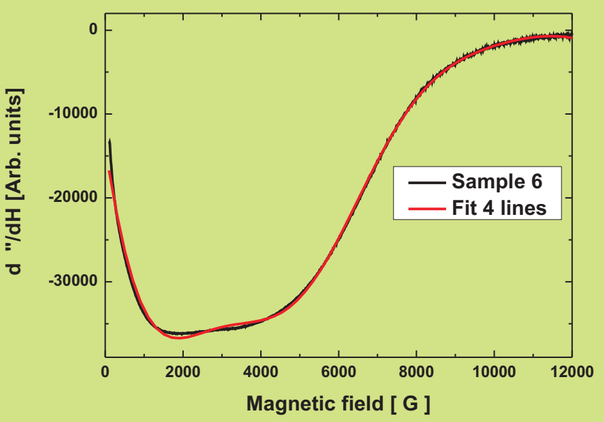












$$I(B) \propto \frac{B_r^2[(B_r^2)+\Delta_B^2](B^2\Delta_B+2B_r|B|\delta_B)+B_r^2(B_r^2+\delta_B^2)\Delta_B}{[(B-B_r)^2B_r^2+(|B|\Delta_B+B_r\delta_B)^2](B+B_r)^2B_r^2+(|B|\Delta_B+B_r\delta_B)^2]}$$

Samples synthesis and characterization

The nitriding process of promoted nanocrystalline iron and the nitrides reduction process at the various nitriding potential (P) on terms of thermodynamic parameters were investigated. During studied processes two parallel reactions occur:

- nitriding reaction, penetration of the chemisorpted atomic nitrogen to crystallographic space in the lattice system of iron (α – Fe(N)), phase transition to iron nitride α' – Fe_4N and next, to ε – Fe_xN; it is observed when critical bulk concentration of nitrogen is reached;
- surface reaction of catalytic ammonia decomposition.

The gas nitriding and reduction were studied in a differential reactor equipped with systems that made it possible to conduct both thermogravimetric measurements ($_{Fe}$) and hydrogen concentration analyser in the reacting gas mixture. The nitriding and reduction processes were investigated under ammonia – hydrogen mixtures (various P), under atmospheric pressure, at 400°C and 475°C. It was found that during the nitriding process as well as the reduction process the stationary states exist, wherein only catalytic ammonia decomposition reaction runs. In the system: nitriding of nanocrystalline iron and reduction of nanocrystalline nitrides for $_{Fe}$ = f(lnP), the hysteresis phenomenon occurs.

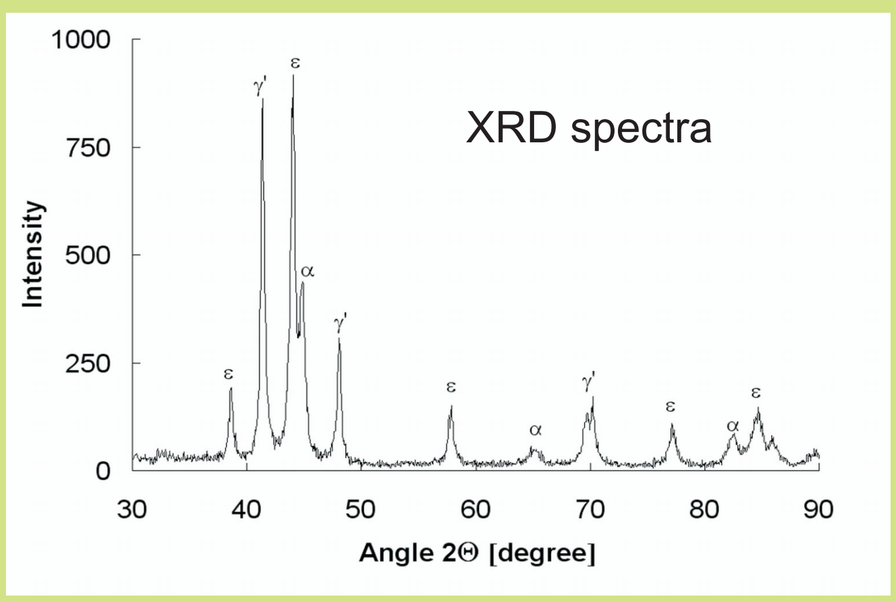
On the basis of XRD studies the areas where phase composition of solid samples was changed during nitriding and reduction process were obtained. It was observed that during nitriding process areas of various phase composition exist in a wide range of nitriding potential:

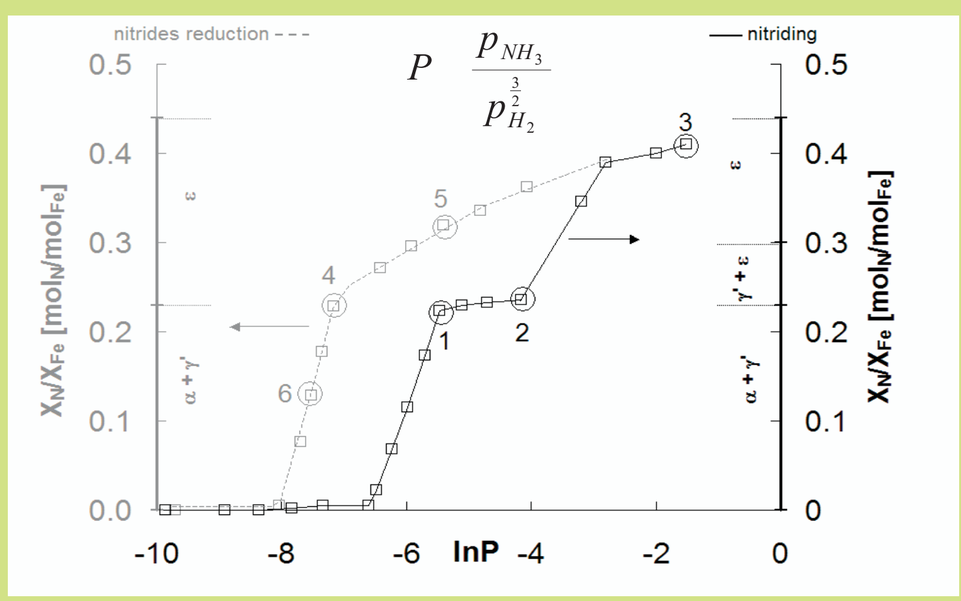
– the observed nitriding degree is relatively low (less than 0.015 mol_N/mol_{Fe}), what corresponds to the nitrogen dissolving and ammonia adsorption on the surface of iron; creation of solid solution of nitrogen in iron (α – Fe(N));

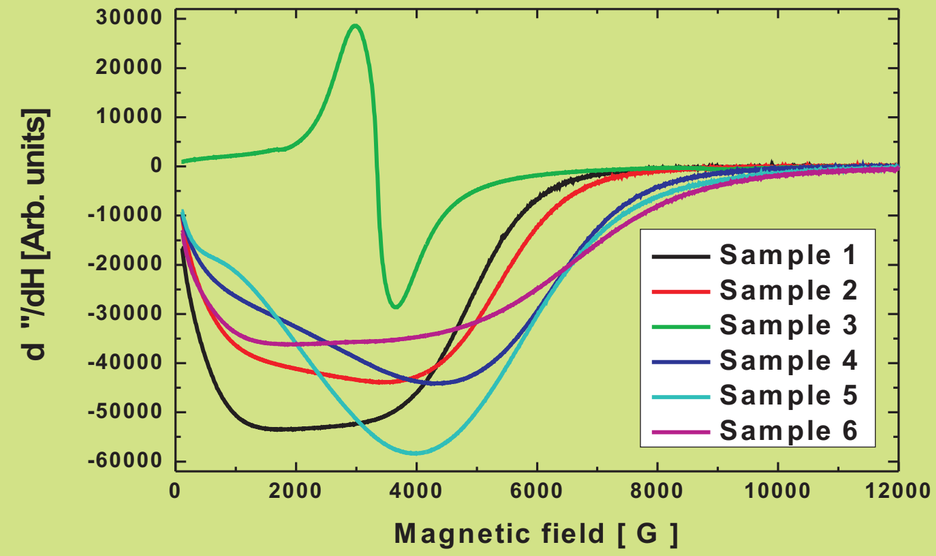
– formation of a new phase thus two phases exist simultaneously in sample: α – Fe(N) and α' – Fe_xN;

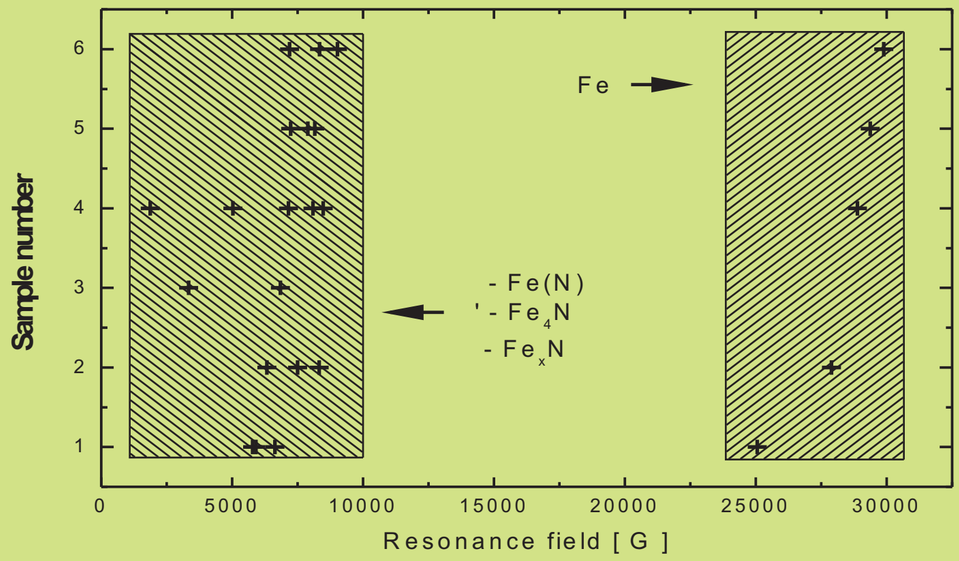
– only one phase exists - α' – $Fe_{4-x}N$, where x decreases with increase of the nitriding potential;

– continuation of saturating of the nitride by nitrogen thus two phases simultaneously exist in sample: α' – Fe_4N and ε – Fe_xN; above that area there is continual saturating of the ε – Fe_xN by nitrogen.









FMR parameters

Sample 1				
Line	1	2	3	4
Amplitude	8.550E11	2.717E14	2.5408E12	3.4468E13
Resonance field	5779.5	25059.6	6637.5	5902.1
Linewidth 1	5625.9	10319.6	2.07E-34	8234.2
Linewidth 2	131.3	20521.1	5245.9	8868.9

Sample 2				
Line	1	2	3	4
Amplitude	1.9846E12	2.7938E14	1.3124E12	3.5893E13
Resonance field	8326.7	27889.9	7506.7	6320.9
Linewidth 1	10965.2	13847.3	1.548E-52	8829.9
Linewidth 2	4.8E-45	18123.4	4537.8	10368.9

Sample 3		
Line	1	2
Amplitude	2.8989E10	5.2674E10
Resonance field	3342.4	6838.6
Linewidth 1	1.6569E-23	8030.7
Linewidth 2	561.2	1.6E-12

Sample 4						
Line	1	2	3	4	5	6
Amplitude	3.0341E12	2.7448E14	1.8519E12	3.1408E13	3.0374E9	2.6495E10
Resonance field	8479.9	28880	8085.1	7145.3	1877.3	5024.0
Linewidth 1	10096	14564	95.2	9894.1	6.727E-49	378.4
Linewidth 2	485.3	16219	5070.2	9790.3	1047.6	1324.9

Sample 5				
Line	1	2	3	4
Amplitude	3.5483E12	2.6986E14	1.8147E12	2.9544E13
Resonance field	8157.2	29367.8	7887.2	7239.0
Linewidth 1	9987.4	14896.7	3.2698E-69	10124.0
Linewidth 2	1038.7	16455.7	5264.9	10215.7

Sample 6				
Line	1	2	3	4
Amplitude	3.4805E12	2.6986E14	1.7852E12	3.1162E13
Resonance field	9020.5	29885.6	8340.7	7190.6
Linewidth 1	9084.5	14774.5	3.2698E-133	9940.3
Linewidth 2	1479.9	17135.9	5863.6	10034.7