IvECHANICAL PROCESSE% IN MAGNETIC MATERIALS

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1. INTRODUCTION

The classification of magnetic materials as soft and hard is based on the magnetic properties. Whereas the soft magnetic materials exhibit a high saturation induction, .a high remanence, a high permeability and a low coercivity, the hard magnets posses a high saturation induction, a high remanence and a very high coercivity. The properties such as remanence, permeability and coercivity are structure-sensitive and hence are affected to a great extent by their microstructures and textures, which in turn depend on the processing methods[1-4]. Soft magnetic alloys which are in great commercial use are the silicon steels and the Ni-Fe alloys. In majority of the applications, these alloys are used in the form of thin sheets or strips. The processing of these alloys includes hot forging, hot rolling, pickling, cold rolling/drawing and suitable heat-treatment. Through judicious selection of the processing parameters including heat treatment, one can evolve the most desirable microstructure or texture.

In the case of magnetic materials, cold work (plastic deformation) makes magnetisation more difficult. The M-H or the B-H curve is lower than that for the fully annealed condition. The hysteresis loop becomes wider (a larger coercivity) and hence a higher hysteresis loss for the same maximum value of M or B. The mechanical strength and hardness also increase. These changes are associated with the increased numbers of dislocations and other lattice defects. The resulting microstress impedes both domain wall motion and domain rotation, increasing the magnetic hardness. Also, the maximum (magnetic) permeability decreases sharply even with a small amount of cold work.

In so far as the permanent magnet alloys are concerned, the powder metallurgical route is the most general method of production. However, deformation aging, swaging; and hot extrusion have been successfully employed to induce 'anisotropy' and hence improved magnetic properties, in some permanent magnet alloys[4,5].

2. FROCFS.SIN3 OF SOFT MAGNETIC AUDYS

Extensive studies have been made by researchers to develop soft magnetic alloys such as low carbon steel, cold rolled non-oriented (CRNO) and cold rolled grain oriented (CRGO) silicon steels and various Ni-Fe alloys with excellent magnetic properties through control of processing parameters. Low carbon steel, which is required for the cores of small 7.otors, has a complex primary recrystallisation texture and is magnetically unattractive. Efforts are being made, therefore, to improve the texture for improving permeability by minor alloying and suitable cc'bination of rolling and heat-treatment.

2.1 **Non-oriented Silicon Steels[1,2]**

Non-oriented silicon steels (containing Si up to 4 wt.%) find applications in small•H.P motors, fans, washing machines, water pumps, refrigerators, energy meters etc. These Si-Fe alloys remain bcc right up to the melting point due to which these alloys can be recrystallised from quite high temperatures, without interference by phase changes, for developing suitable texture.

There are two standard ways of producing non-oriented silicon steels. After hot forging and hot rolling (to 1.8-2.5 mm thickness), the sheets are pickled and either cold rolled to a final thickness (0.35-0.5 mm) and then strand annealed or cold rolled to an intermediate thickness (of 0.5-0.6 mm) strand annealed, cold rolled again slightly to the final thickness and again strand annealed. CRNO steels have an optimum grain size of ASTM 5-3 (0.05 mm). They exhibit a core loss of 2-5 watts/kg. The control of cold reduction is essential for reducing the core losses.

2.2 Cold Rolled Grain Oriented Silicon Steel[1,2]

These steels contain about 3.0-3.25 wt.% Si. Generally the steel sheets are of thickness 0.15-0.35 mm. These are widely used in power and distribution transfor7ers.

Thermomechanical processing stages are so designed that a highly textured material is produced. Cold rolling with intermediate anneals, plus a high temperature anneal produce sheets with much better magnetic properties in the rolling direction than the hot rolled sheet. This improvement is due to preferential orientation (texture) of grains or crystals produced by secondary recrystallisation during high temperature annealing.

There are two types of textures in silicon steels, namely (i) Cube-on-edge, (110)[001] and (ii) Cube, (100)[100] textures. In the cube-on-edge texture, the (110) planes are parallel to the plane of the sheet and that the [001] directions in these planes are parallel to the rolling direction. As the name itself suggests, the cube is lying on its edge with the cube edge parallel to the rolling direction and the diagonal (110) plane parallel to the rolling plane. In the cube texture, the (100) planes are parallel to the plane of the sheet and the [100] directions are parallel to the rolling direction. Generally, cube-on-edge textured material is produced. By producing a nearly perfect cube-on-edge texture (with average deviation not more than 3-4°), the core loss of the sheets can be considerably reduced (< 0.4 watts/kg at 1.5 Tesla).

The processing of CRGO steels comprises of the following steps:

(a) Hot rolling the cast ingot (containing 3.2% Si, 0.01-0.02% C, 0.06-0.10% Mn and 0.02% S) at about 1300°C to a thickness of 1.5-2.0 mm.

(b) Removing the oxide scale by acid pickling.

(c) Cold rolling to a final thickness of 0.15-0.35 mm in two approximately equal steps, with an intermediate annealing at 800-1000°C (The total cold reduction in thickness should be of the order of 85%).

(d) Decarburizing at 800°C in moist hydrogen. This annealing reduces the carbon level to below 0.003% and causes primary recrystallisation.

(e) Annealing in dry hydrogen at 1100-1200°C to develop the cube-on-edge texture by secondary recrystallisation.

In the sheets having cube-on-edge texture, the magnetic properties in the rolling direction are excellent whereas that in the other directions are inferior. The optimum grain size is of the order of 4-7 mm. Currently, there has been great interest in reducing the core losses through production of still thinner gauge steels.

2.3 Nickel-iron Alloys[2,3,6]

These are alloys containing 36-80% Ni and balance iron (with or without minor additions of other elements). The different Ni-Fe alloys are known by their trade names as permalloys (50-80, Ni-Fe), Mu metal (75% Ni, 5% Cu, balance Fe with or without 2% Cr), Rho metal (36-42% Ni-Fe), radio metal (45-50% Ni-Fe) and HCR alloys (45-50% Ni-Fe). In the form of stacked laminations or tape wound cores or powder cores, they find applications in loading coils, special transformers (in communication equipment), magnetic amplifiers, sensitive relays, twistor memories, shielding applications etc.

Processes for the production of different types of Ni-Fe alloys similar to Mu metal, radio metal, Rho metal and HCR alloys have been developed at NML, Jamshedpur. The processing of these alloys may be Summarized as follows:

- Melting nickel and iron in vacuum/air and casting,
- Hot forging followed by hot rolling at 1100'C to a thickness of 2.5 mm,
- Pickling in dil. H_2SO_4 to remove the oxide scale,
- Cold rolling, without intermediate annealing, giving a thickness reduction of 90% (and maintaining the reduction in each pass at 10-15%), and
- Heat-treating at 1000-1200°C in pure, dry hydrogen atmosphere.

The magnetic properties of various Ni -Fe alloys developed at NML, are given in Table 1.

Table 1: Properties of Ni-Fe alloys developed at NML

Alloy	Permeability		Saturation induction (gauss)	Coercive force (Oe)	Electrical resistivity (micro ohm cm)
	Initial	Maximum			
Mu metal	35,000-45,000	80,000-2,25,000	6,000-7,200	0.01-0.02	
Rho metal		12,000-30,000	11,000-14,000	0.08-0.14	69-90
Radio metal		30,000-45,000	14,000-15,000	0.06-0.16	45-50
HCR alloy		30,000-48,000	14,000-15,500	0.07-0.20	

It is possible to produce cube textured Ni-Fe alloys at or near the 50-50 composition by a large amount of cold rolling (95% or more) followed by annealing in dry hydrogen at temperatures above 800°C. The orientation (010)[100] is formed by primary recrystallisation. Such oriented materials exhibit square hysteresis loops and are used in tape wound cores.

2.4 High Strength Soft Magnetic Alloys for High Speed Rotors[7,81

With a view to developing high strength materials with moderately soft magnetic properties for applications in hydrogenerator rotor poles and rotors of portable generators, extensive studies were carried out at NML on modifying the properties through microstructural manipulation, in some commercially available structural steels.

Commercial grade HSLA steel in the form of hot rolled plates of thickness 8.0 mm having the composition C 0.22, Mn 1.77, V 0.12, Nb 0.04 and Ti 0.04 balance Fe (all in wt.%) was chosen for the study. The object of the study was to improve the magnetic properties without impairing the mechanical properties, through spheroidisation of carbides[9-12]. Accordingly, the material was processed through the following TMT (thermomechanical treatment) routes:

(i) hot rolling from 1130°C and finish rolling at 800°C and quenching to room temperature followed by tempering at 675°C for duration of up to 52 h (RQT),

(ii) hot forging at 1150-800°C, quenching and tempering at 675°C for duration of up to 52 h (FQT).

The TMT routes were followed in order to accelerate spheroidisation of carbides. Fig.1 shows the microstructure of steel in the as received condition. Figs.2 and 3 show the microstructures of RQ and RQT, and FQ and FQT samples. The steel quenched from 930°C shows acicular ferrite, lath martensite and precipitated carbides in ferrite. On tempering, the martensite breaks up into ferrite and carbides during the initial stages and with increase in tempering time, the growth of carbides and eventual spheroidisation and coarsening results. The mechanical and magnetic properties of the steel after different treatments are given in Table-2. Both the RQT and FQT samples have shown, after tempering at 675°C for 30 h, a reasonably good combination of strength and magnetic properties.

3. PROCESSINU OF PERMANENT MAGNET ALLAYS

Most of the permanent magnets in use today are made either by powder metallurgical processing or through melting, casting and heat treatment[4]. However, in alloys such as Mn-Al-C (containing near equiatomic percent of Mn and Al and about 0.5 wt.% C) and Fe-Cr-Co alloys (containing 10-15 wt.% Co, 28-33 wt.% Cr, small amounts of Nb and Al and balance Fe) improved (anisotropic) permanent magnet properties can be realised through cold working and heat-treatment or through hot extrusion[5,13-15].

For making >!n-Al-C alloys anisotropic, the alloy billet is solutionised at -- 1100°C for 1 h, quenched to N 500°C and then tempered at 600°C for 0.5-1.0 h. This treatment is required as the high temperature phase which is non-magnetic changes over to the ferromagnetic fct phase on tempering. The alloy billet is then extruded using a hydraulic press at 700°C at a pressure of 70-80 kg/mm^2, giving an area reduction ratio of ~7.0. Fig.4 shows the mechanism by which the alloy becomes anisotropic after extrusion. Because the alloy is hard and brittle, the deformation process is quite difficult. The results of the studies carried out at NML (using extrusion facilities available at NPL, Delhi) are given in Table-3. The improvements in the properties as compared to that of isotropic material is only marginal and this is attributed to insufficient deformation that could be given to the material at the extrusion

Table 2: Mechanical and magnetic properties of HSLA steel after thermomechanical treatment

Prior treatment	[illegible]	[illegible]	Elongation (%)	Induction at 95 Oe (Tesla)	Remanence, Br, (Tesla)	Coercive force, Hc, (Oersted)	Permeability u max
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]

[illegible] Hot forged + [illegible]

temperature of only 400°C. Probably better area reduction, and hence better anisotropy, could have been possible had the extrusion temperature been maintained around 600-700°C.

Table 3: Magnetic properties of Mn-Al-C permanent magnets

Alloy composition	Treatment given	Magnetic properties		
		Remanence (gauss)	Coercive force (Oe)	Maximum energy product (MG Oe)
Mn 69.2, Al 29.2, C 0.53, Ni 1.0 (all in wt.%)	Tempered at 600°C after quenching from 1000°C	2450	1020	0.9
Mn 69.2, Al 29.2, C 0.53, Ni 1.0 (all in wt.%)	Extruded at 400°C + aged at 320°/ 45 mts	3550	710	1.04
Mn 68.6, Al 28.8, C 0.85, Ni 2.0 (all in wt.%)	*Extruded at 400°C + aged at 320°/ 45 mts	4150	780	1.40

* Extrusion ratio 6.0, Extrusion pressure 3000 psi, Reduction of area 15%.

Fe-Cr-Co alloys like the Alnicos are spinodal alloys and exhibit permanent magnet characteristics. The isotropic alloys, however, are unattractive because of their low coercivity and low maximum energy product $(BH)_{max}$. The isotropic alloys, however, are unattractive because of their low coercivity and low $(BH)_{max}$. At cobalt content above 15 wt.%, they can be made anisotropic (shape anisotropy) through step tempering treatments. However, low cobalt (10-15 wt.%) containing alloys do not show marked anisotropic behaviour after step tempering. Since these alloys are more ductile as compared to high cobalt containing alloys, deformation aging can be employed to induce shape anisotropy and thereby improve coercivity and $(BH)_{max}$. One of the processing schedules employed at NML for developing anisotropy is given in Fig.5. The microstructures of the step tempered (isotropic) and deformation aged alloy (anisotropic) are shown in Fig.6. The magnetic properties of these alloys are given in Table-4.

Table 4: Magnetic properties of Fe-Cr-Co alloys

Alloy composition	Remanence, Br (gauss)	Coercive force, He (Oersted)	Maximum energy product, (BH) (MG 8g
Cr 33, Co 14, Nb 1 balance Fe (isotropic)	8,440	455	1.06
*Cr 33, Co 14, Nb 1 balance Fe (anisotropic)	10,660	580	3.42
Cr 33, Co 12, Nb 1 balance Fe (isotropic)	7,700	445	1.20
*Cr 33, Co 12, Nb 1 balance Fe (anisotropic)	8,970	620	2.70

* deformation aged alloys

4. CONCLUDING RDIARKS

Mechanical processing is an important step in the production of magnetic materials, in particular, the soft magnetic ones. The control of microstructure -- the grain size and shape and distribution of the second phase particles -- is vital for enhancing the magnetic properties. In some materials like silicon steels and some Ni-Fe alloys, development of suitable texture through controlled mechanical processing and heat treatment is very much essential in order that they are well suited for the applications. Also, the surface finish of the rolled sheets, is important, for lany applications. In the case of some permanent magnets, though hot or cold deformation processing induces anisotropy and improves magnetic properties, magnets processed through these routes are probably not available in market even today.

REFERENCES

1. R.M.Bozorth, Ferromagnetism, D.Van Nostrand, New York, 1951.

2. B.D.Cullity, Introduction to Magnetic Materials, Addison Wesley, 1972.

3. J.K.Stanley, Electrical and Magnetic Properties of Metals ASM (1963).

4. M.NtCaig, Permanent Magnets in Theory and Practice, Pentech Press, London (1977).

5. T.Kubo, T.Ohtani, S.Kojima and N.Kato, JEE, No.127 (1977) 50

6. G.Y.Chin and J.H.Wernick, Ferromagnetic Materials, Vol.2, p. 55-188, Ed. E.P.Wohlfarth, North-Holland (Amsterdam), 1980.

7. S.P.Narayan, V.Rao, S.Das and 0.N.Mohanty, J.Magn.Magn.Mat., Vol. 88 (1990) 71.

8. S.P.Narayan, V.Rao and 0.N.Mohanty. J. Magn.Magn.Mat., Vol.99 (1991) 137.

9. A.T.English, Acta Met., Vol.15 (1967) 1573.

10. J.H.Swisher, A.T.English and R.C.Szoffers, Trans. ASM, Vol.62 (1969) 257.

11. D.C.Jiles, J.\ppl.Phys, Vol.63 (1988) 2980.

12. D.C.Jiles, J.Phys.D, Vol.21 (1988) 11E6.

13. Ya S.Shur, Ye V.Belozerov, L.M.Magat. Phys.Metals and Metallography, Vol.47(5) (1979) 37.

14. T.S.Chin, T.S.Wu, C.Y.Chang, T.K.Hsu aDd Y.H.Chang, J. Mat. Sci, Vol.18 (1983) 1681.

15. S.Jin, S.Mahajan and D.Brasen, Met.Trans (A), Vol.11A (1980) 69.

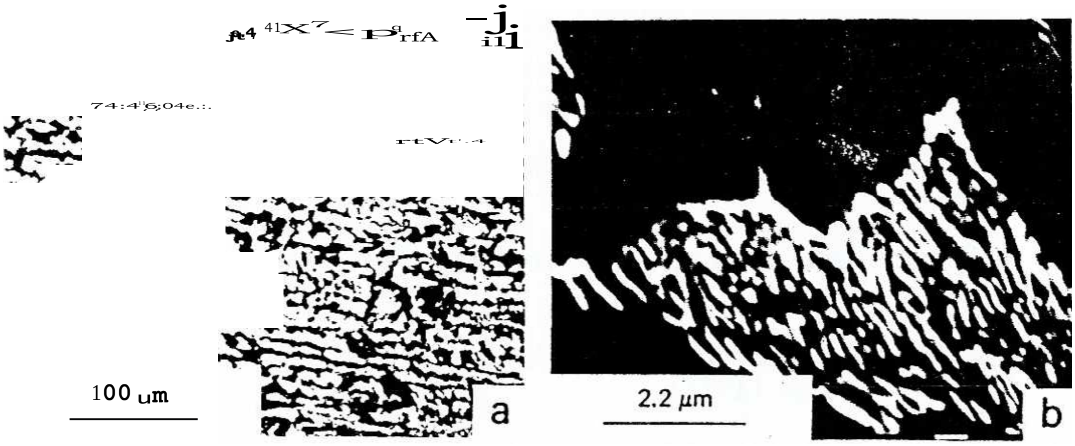


Fig.1. Microstructures of HSLA steel in as-received condition.
a) Optical micrograph showing banded structure. b) SEM micrograph showing cementite lamellae in pearlite colony.

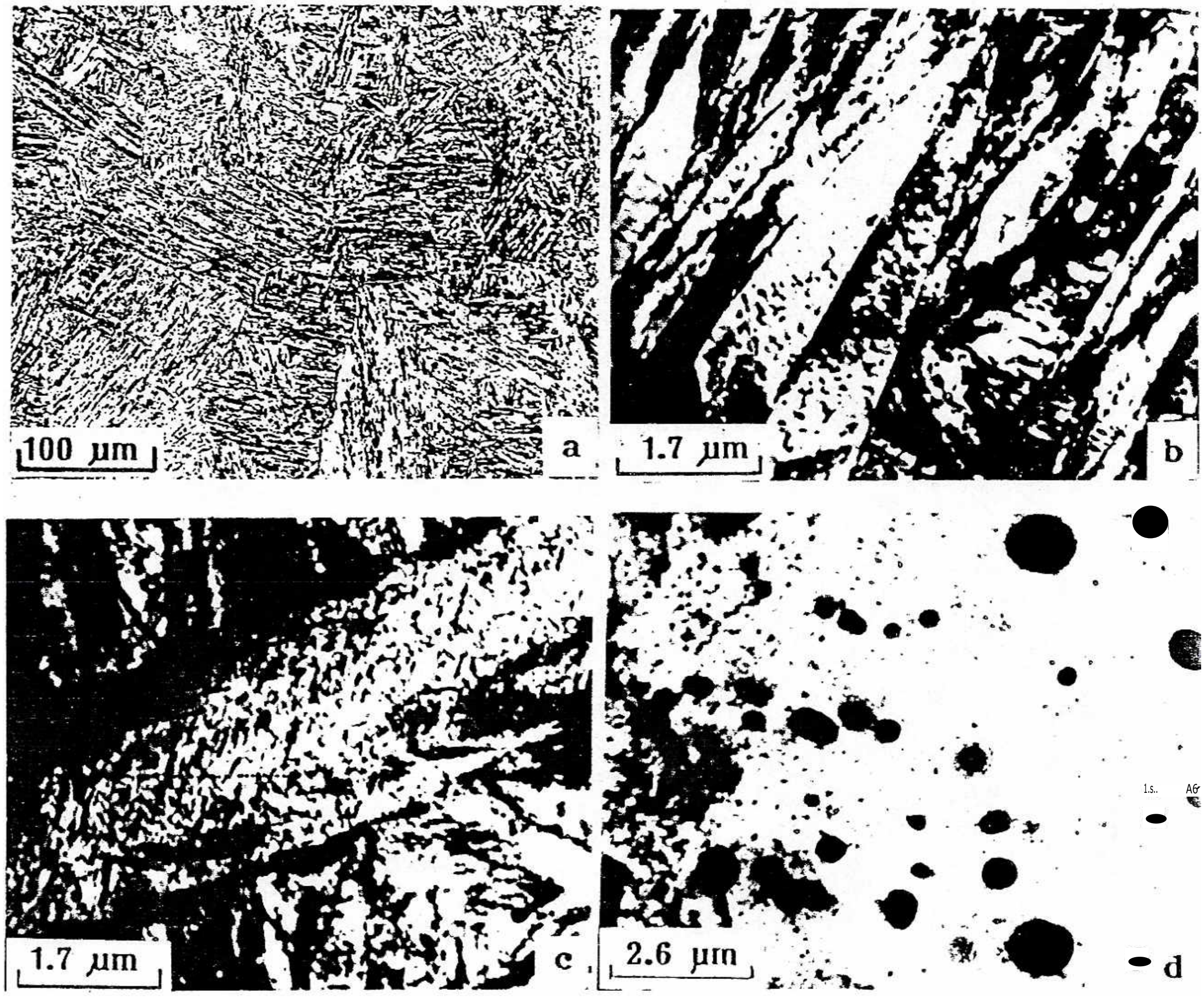


Fig.2. a -c) Microstructures of Rolled + water quenched (RO) and d) Rolled + water quenched + tempered at 675°C for 52 hrs. (ROT), HSLA steel. Fig. (a) shows needle like features. Figs. (b) and (c) are TEM micrographs showing acicular f errite, lath martensite. Fig. (d) is the TEM micrograph showing spheroidised carbides.

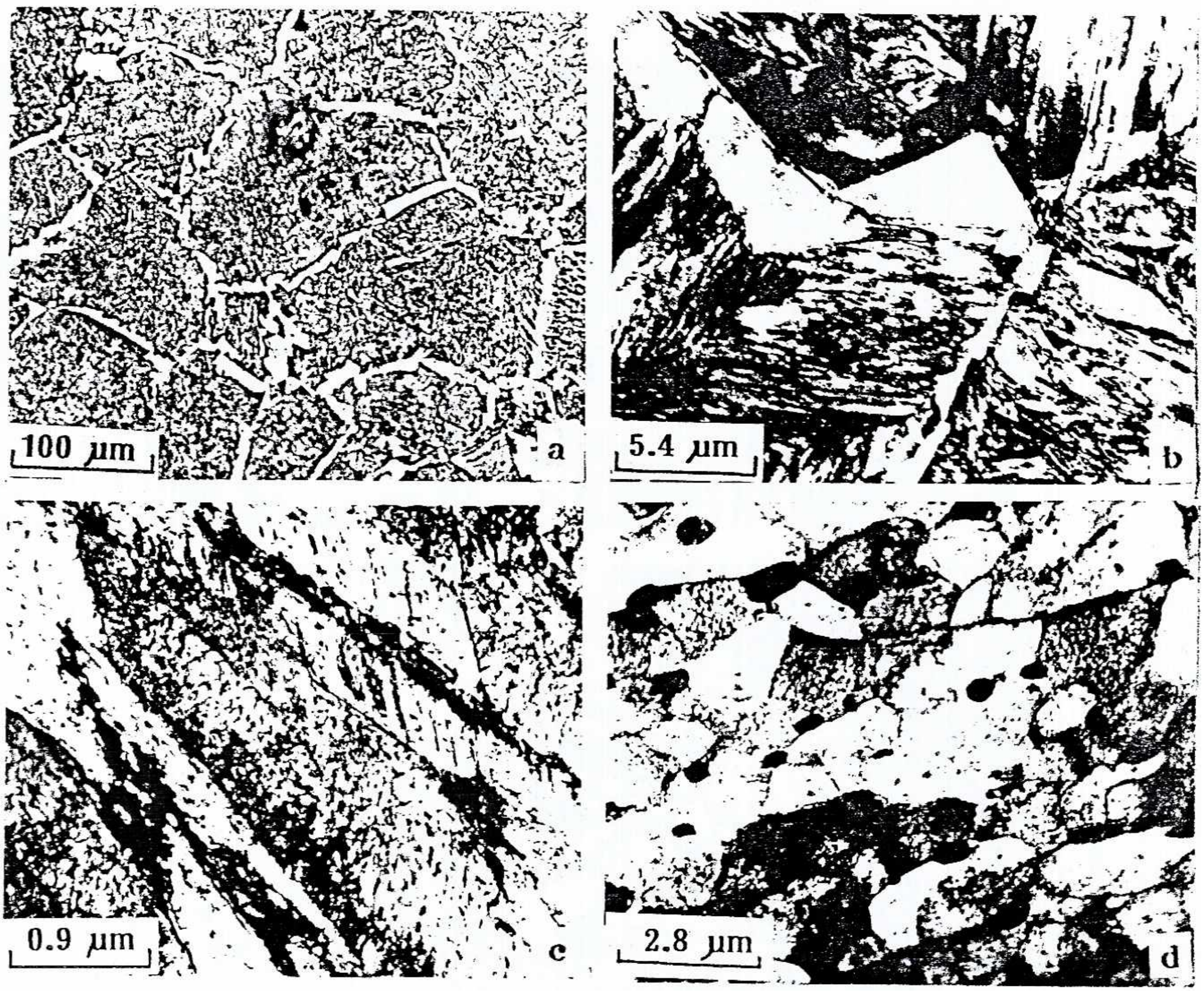


Fig.3. (a)-(c) Microstructures of Forged + water quenched (FQ) and (d) Forged + water quenched + tempered at 675 °C for 52 hrs.(FOT), HSLA steel. Fig. (a) is the optical micrograph showing thick ferrite along the prior austenite grain boundaries. Figs. (b) and (c) are the TEM micrographs showing slabs of ferrite precipitation, lath martensite and acicular ferrite. Fig.(d) shows spheroidisation of carbides and polygonisation of ferrites.

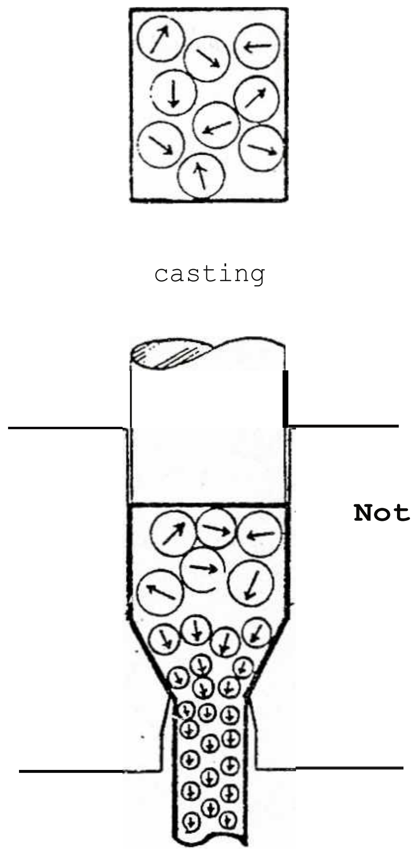


Fig.4. Not Extrusion of Mn-Al-C alloy makes it anisotropic. Spin magnetic moments orient in a parallel way.

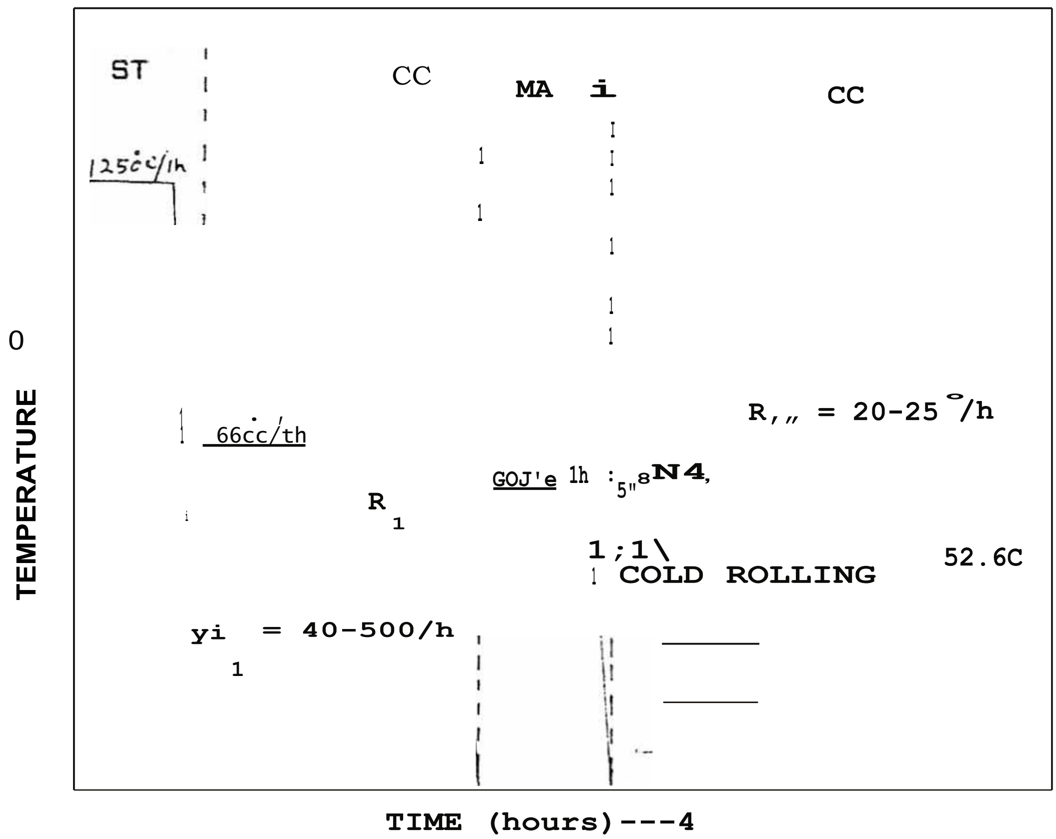
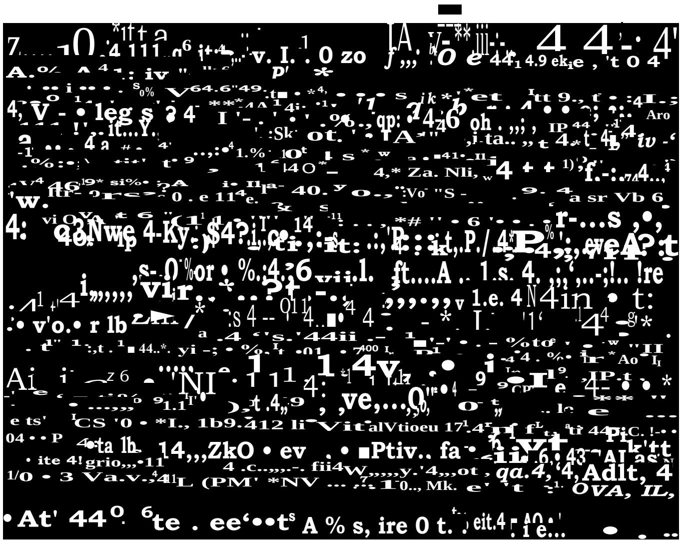


Fig.5. Deformation aging process for making Fe-Cr-Co alloy anisotropic. (ST = Solution treatment, CC =Control Cooling, MA = Magnetic Annealing)



(a)

(b) *X5.koco*

Fig.6. TEM micrographs of a) Solution treated and aged Fe-Cr-Co alloy (no deformation given) b) Solution treated and deformation aged Fe-Cr-Co alloy, showing elongated grains. (ST = Solution treatment, CC = Control Cooling, **MA =** Magnetic aging).