Nanocrystallinity and enhancing magnetism in soft Fe-Si-Nb-Cu alloy

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Introduction

•Fe-Cu-Si-Nb-B alloy discovered in 1988[1]

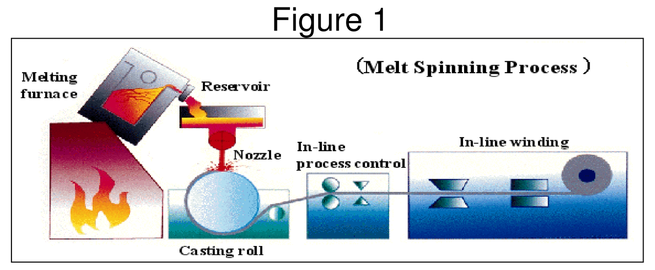
•Applications in perpendicular recording media, transformers etc.[4]

•Nano-grains can be produced by annealing, improving soft magnetic properties:
- -high saturation magnetization
- -high permeability
- -low energy loss

•This study aimed at developing enhanced magnetic properties through annealing and controlled atmosphere

Fabrication by rapid solidification (melt-spun quenching)

Figure 1

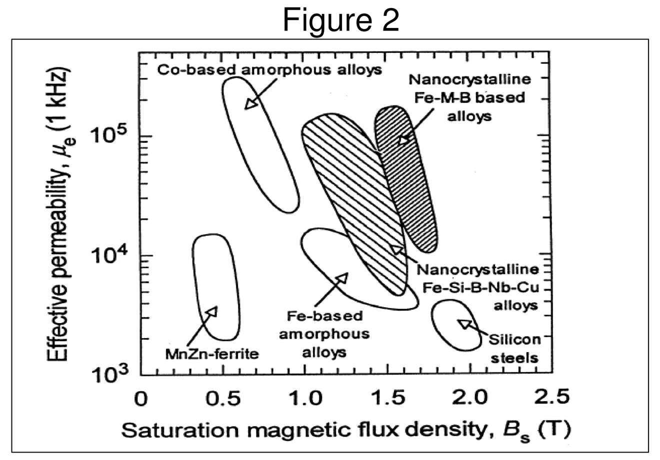


Courtesy Hitachi Metals

1. Melt cooled by pouring onto a spinning wheel
2. Alloy cools rapidly; inhibits equilibrium processes
   -Amorphous material forms
3. Measurements (Figure 2) show relatively high magnetic permeabilities and high saturation magnetization compared to other alloys
4. Heat treatment process (annealing) allows for nano-crystals to form

Alloy exhibits good permeability, saturation magnetization vs. other alloys (Figure 2)

Figure 2



Courtesy www.wtec.org[2]

Experiment

•Ribbons annealed for temperatures T > 500° C

•Vibrating Sample Magnetometry (VSM) measures magnetic properties

•X-Ray Diffraction (XRD) determines crystalline properties

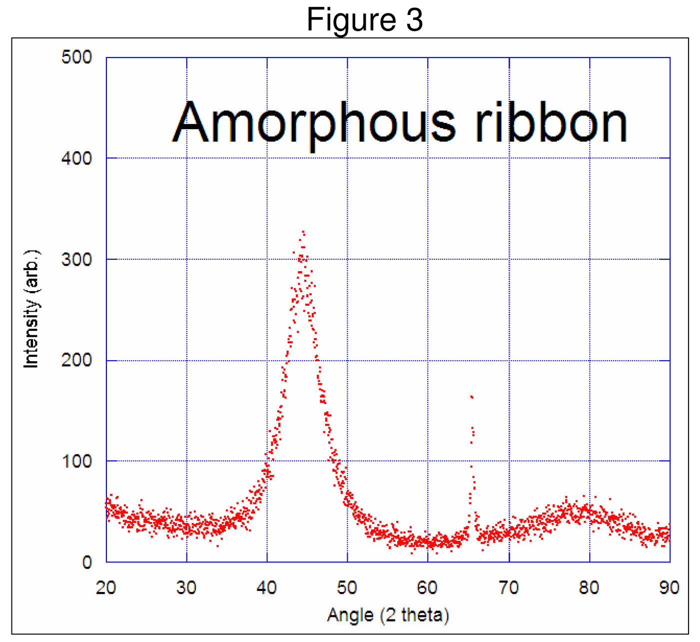
•Scanning Electron Microscopy (SEM) probes structural transitions and determines morphology

Results

X-Ray Diffraction

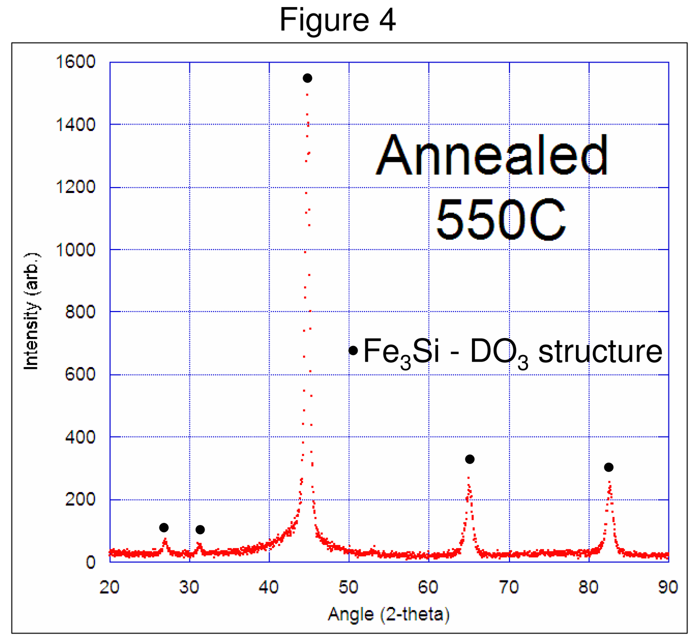
•XDS-2000 XRD used with Cu anode λ = 1.54 Å

Figure 3



•Annealed 550° C sample XRD pattern shows crystallinity in sharp and narrow crystalline peaks:

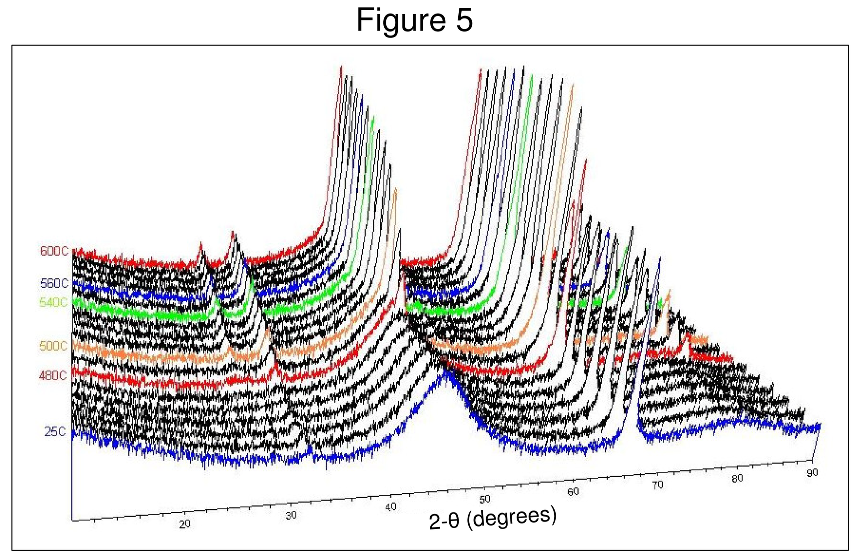
Figure 4



•Temperature-dependent XRD shows evolution of nanocrystalline state in alloy (Figure 5)

(note colored patterns correspond to colored loops in Figure 9, right)

Figure 5



Vibrating Sample Magnetometry

- External magnetic fields (H) allow for measurable magnetization B (= H + 4πM)
- Hysteresis loops obtained from VSM. Loops contain many critical points (Figure 6)

Figure 6 – Typical Hysteresis Loop

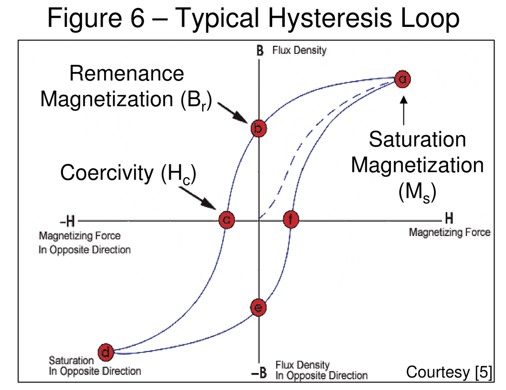
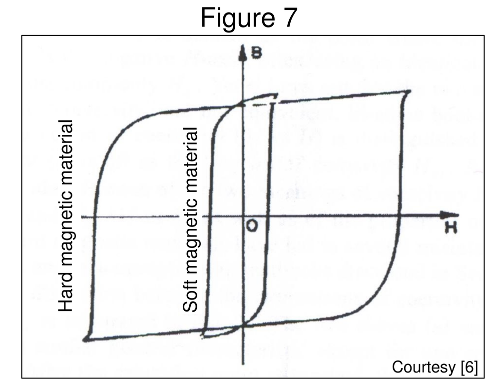


Figure 7

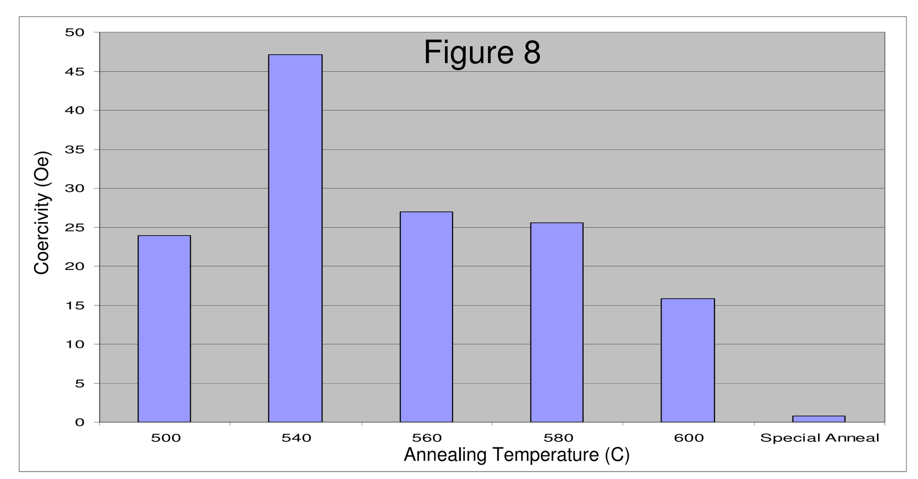


•Area within hysteresis loop corresponds to energy to switch magnetic moment of material (Figure 7)

⟶ Fe alloy has very thin hysteresis loops, very little energy to change magnetic moment

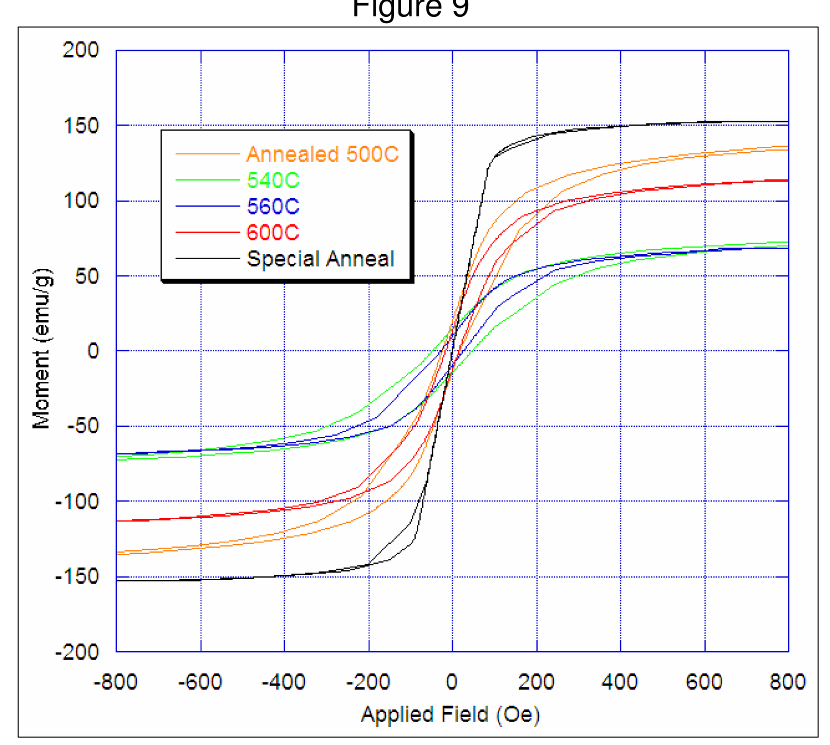
⟶ Permanent magnets give broad loops, much energy to change magnetic moment

Coercivity dependence on temperature shown (Figure 8)



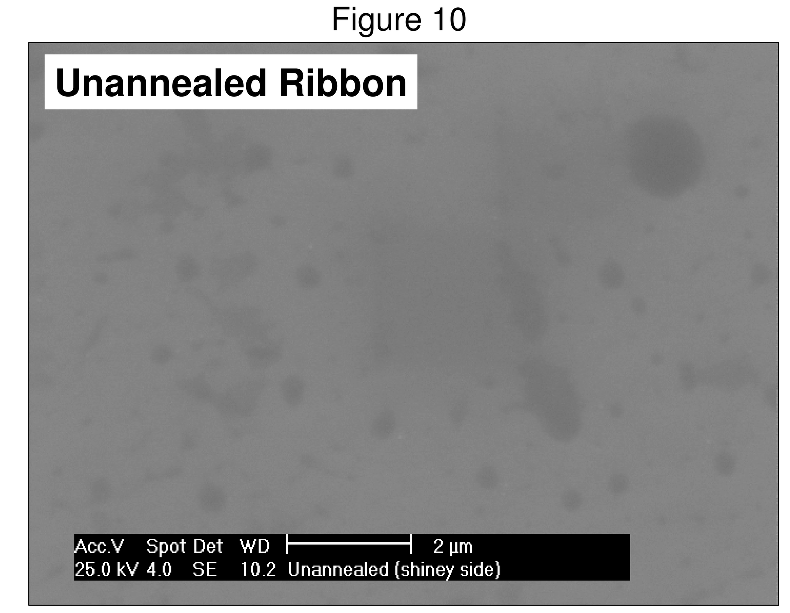
•Special annealing condition (proprietary) further enhances magnetic properties (Figure 9)

Figure 9



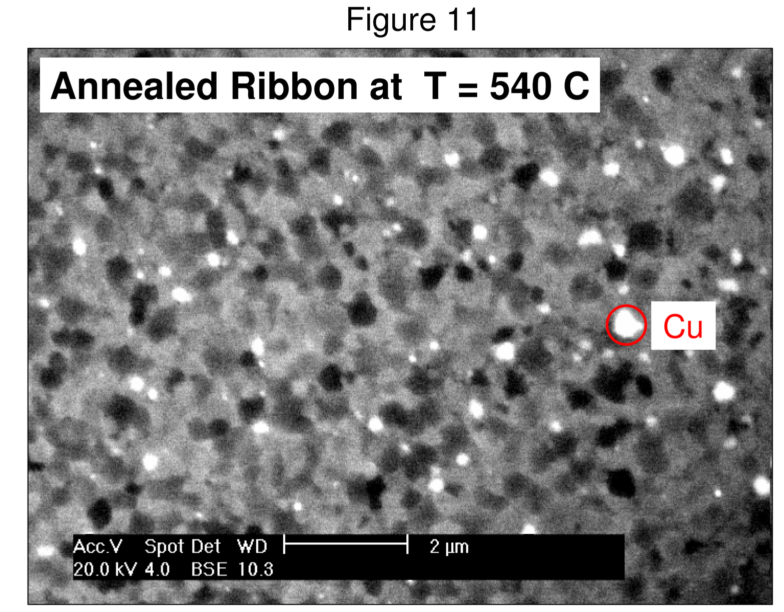
Scanning Electron Microscopy

Figure 10



No crystallinity observed - **Ribbon is Amorphous**

Figure 11

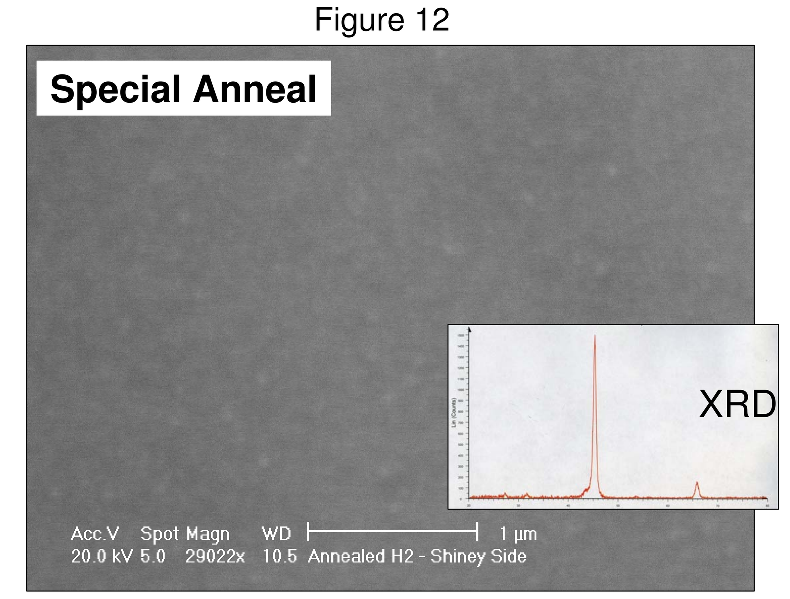


Significant crystallinity!!!

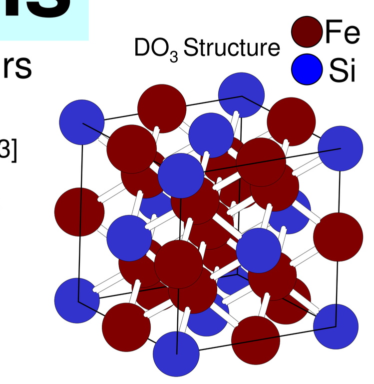
Cu concentrated regions → bright spots as labeled (Figure 11)

•Special Anneal
- -SEM lacks resolution to image ~10 nm grains
- -Alloy appears amorphous, but crystalline character revealed in XRD (Figure 12)

Figure 12



Conclusions



•Primary crystallization occurs at T_1 = 480C
- -Fe_3Si DO_3 crystals form[3]
- -Soft magnetic properties enhanced

•Secondary crystallization occurs at T_2 > 600C
- -Fe_2B and/or Fe_3B phase appears, inhibits soft magnetic properties[4]

•Special annealing gives greatly enhanced magnetic properties
- -Coercivity greatly decreases
- -Saturation magnetization increases

•Superparamagnetic effect may help explain enhanced magnetic properties
- -Small grain size (< 10 nm) necessary
- -Dipole-dipole interaction (~$1/r^3$) between grains yields thermally driven fluctuations of magnetic moments
- -Thermal fluctuations suppress magnetization, lowers coercivity and energy to realign moment.

Future Work

•Further development of special anneal study
- -Understand cause for dramatic enhancement

•Examine new possibilities for annealing conditions

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