

Synthesis of Nanocrystalline
Materials using Electro-
deposition Techniques.

By:-

Ravi Shankar S.
Saurabh Roy
Anand Gopal Datta
Swagatika Das

Guided By:-

Prof. I. Manna
And
Mr. Anindya Basu

Introduction:

Definition:- Nanocrystalline materials are polycrystalline materials with grain sizes of up to about 100 nanometers.

Because of extremely large surface area to volume ratio these materials have some special properties over usual coarse grained materials.

Preparation:-

- **Inert Gas Condensation**
- **Ball milling**
- **Plasma implantation**
- **Electrodeposition**
- **And many others**

Properties:- Nanocrystalline materials exhibit

- **increased strength/hardness,**
- **enhanced diffusivity,**
- **improved ductility/toughness,**
- **reduced density elastic modulus,**
- **higher electrical resistivity,**
- **increased specific heat,**
- **higher thermal expansion coefficient,**
- **lower thermal conductivity and**
- **superior soft magnetic properties**

Electro deposition

- **A coating application method in which metal substrate, an electrical conductor, is made one of the electrodes; the other electrode is generally a metal.**

We have tried to prepare nanocrystalline materials by direct current.

- **The requirements for Nanocrystalline deposition are:**
 - **High current density (usually high current density favors formation of nano crystals.)**
 - **Lower temperature**
 - **Constant stirring for uniform deposition**
 - **Additives**

Objectives:-

- 1) To prepare nanocrystalline materials by electro deposition using direct current.**
- 2) To study and characterize the effects of an electro deposition of nanocrystalline particles of Nickel and Iron on Copper substrate.**
- 3) To study and characterize the layered deposition of Nickel and Iron on Copper substrate.**
- 4) To create a pulse modulated alloy of Ni and Fe on a copper substrate.**

Experiments:

Since we did not have a pulse generator we tried to adopt an indirect route to develop modulated alloy. On the copper substrate we deposited Ni over which a separate layer of Fe was deposited, this was heated to produce a modulated alloy.

On a copper substrate we deposited Ni and Fe separately using various time and current densities and used the optimum combination for further work.

Nickel Bath:

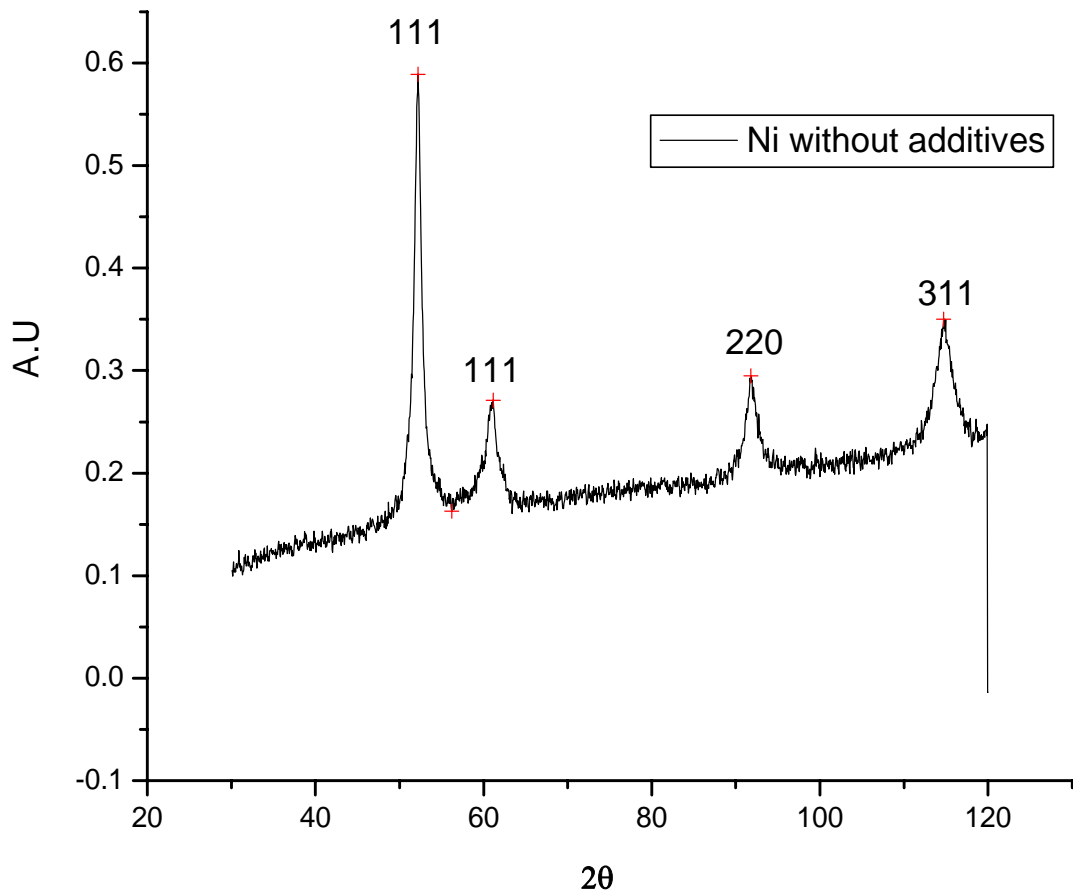
Constituents	Amounts (g/l)
Nickel Sulphate Hexahydrate	350
Nickel Chloride	45
Boric Acid	37

Iron Bath:

Constituents	Amounts (g/l)
Ferrous Chloride Tetrahydrate	300
Calcium Chloride	335

- Vickers micro hardness tests of the sample was done.
- Heat treatment was done on the sample so that alloy may form by diffusion, the results will be added with continued studies.

XRD- Results

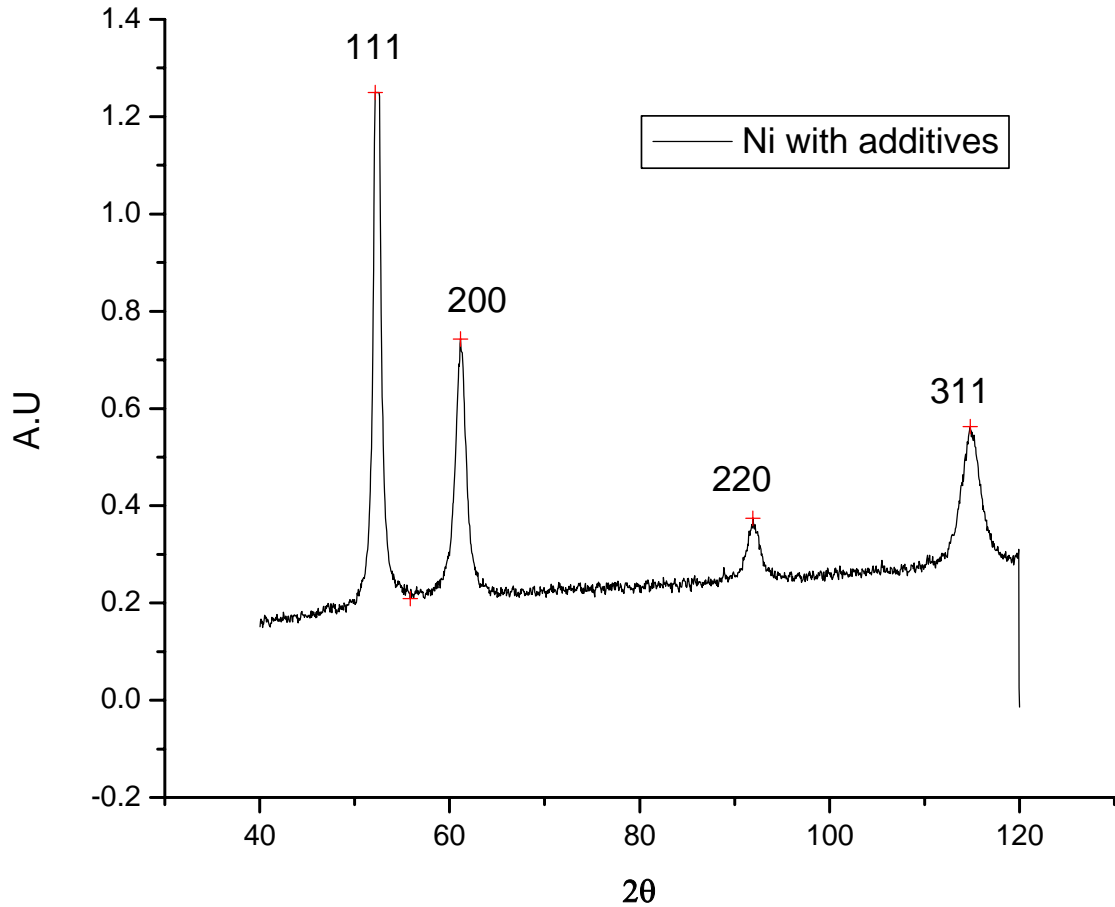


Current Density: 0.1amp/cm²

Size of grains: 97nm

Strain: 17%

Time: 15 min



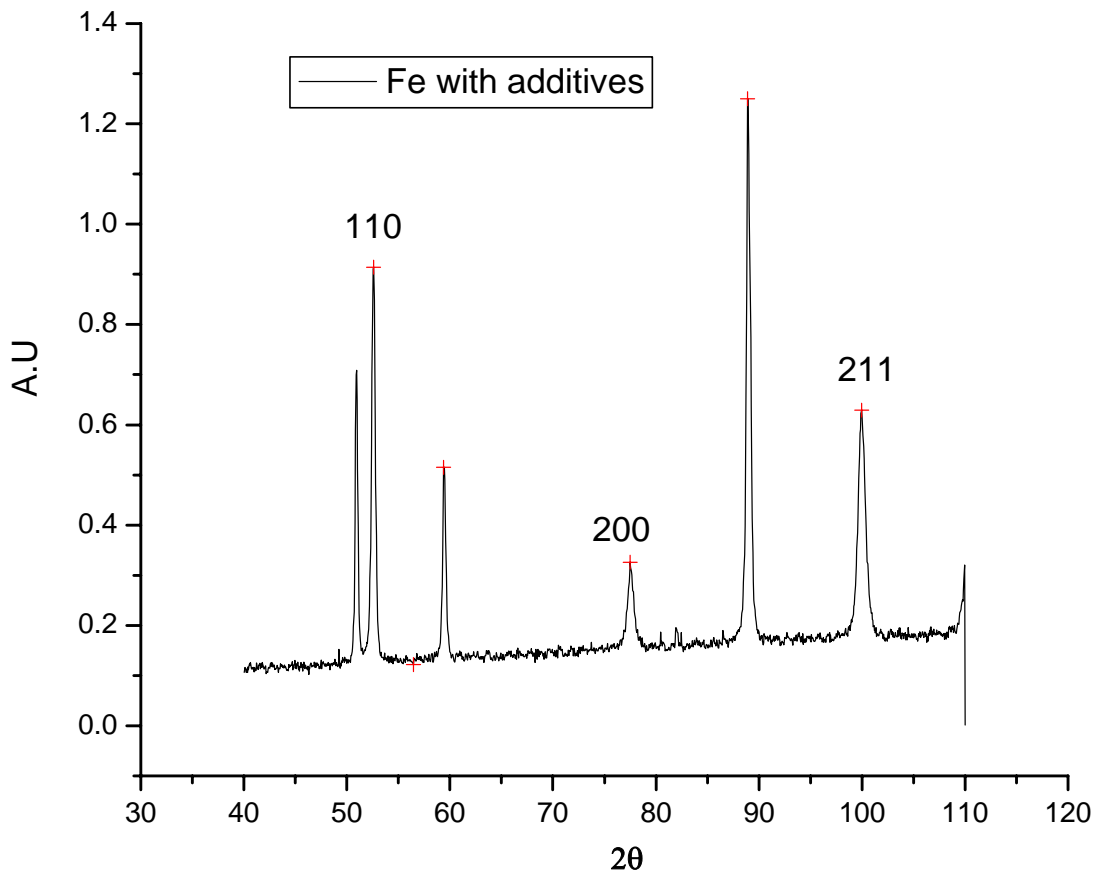
Current Density: 0.1amp/cm²

Grain Size: 37nm

Strain: 37%

Time: 15 min

In Ni we find that there is a definitive reduction in the grain size of the deposited metal. The standard ST-CRY software was used to calculate the grain size of the deposit.



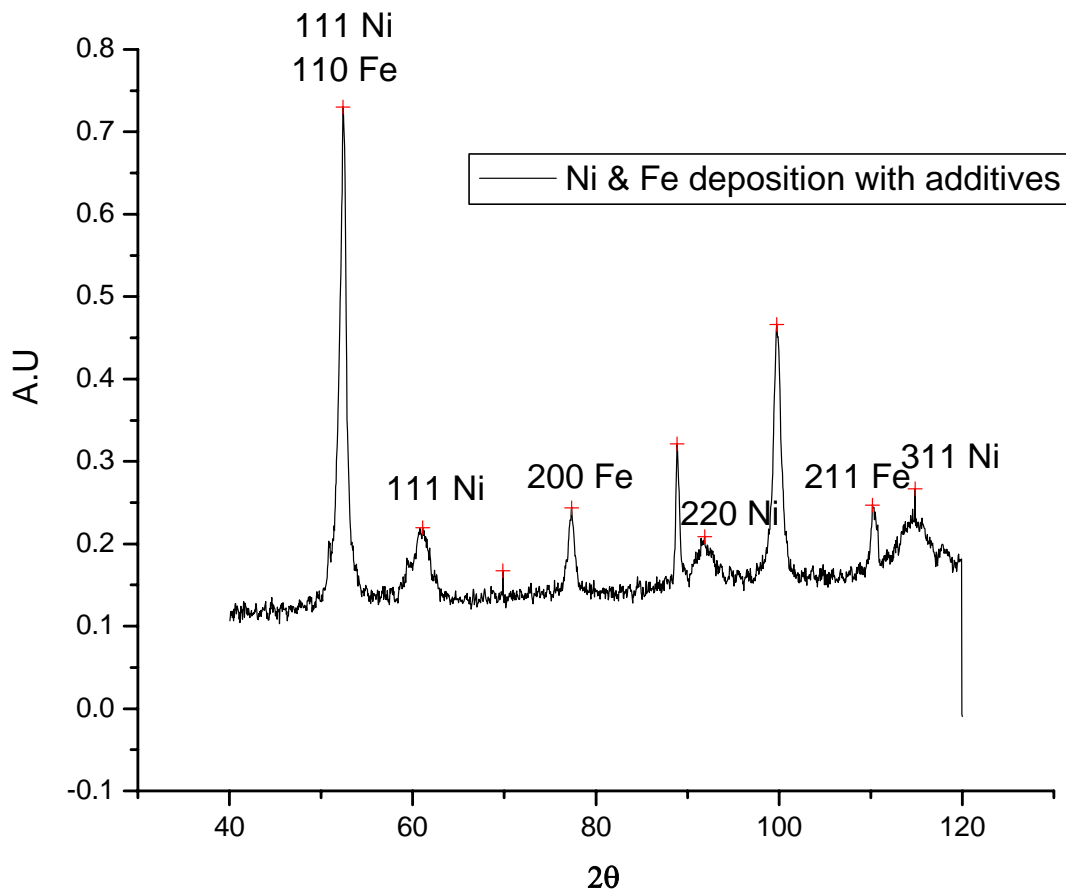
Current Density: 0.1amp/cm²

Grain size: 90 nm

Strain: 23%

Time: 15 min

In case of Iron no difference was found between the deposition with and without saccharine.



Alternately deposited layers of Nickel and Iron on copper substrate. Layer thickness is of the order of 5 μm . The layer thickness was much more than what we had targeted which we hope to reduce in further work.

Results:

- 1) We were successful in our attempt to create nano sized grains by direct deposition.**
- 2) Saccharin which acts as a stress reliever was crucial in our experiment as it relieved the stress in the deposits making smaller grain size possible.**
- 3) Not much difference in the hardness was found in the original sample before and after deposition. This is because we took micro hardness and we needed to take nano hardness, which shall be done in further work.**