



INTERNATIONAL JOURNAL OF CREATIVE RESEARCH THOUGHTS (IJCRT)

An International Open Access, Peer-reviewed, Refereed Journal

CHARACTERISATION OF NEW PEAKS IN XRD ELECTRO-POLISHED IRON PREPARED BY VACCUM ARC MELTING

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ABSTRACT: X ray Diffraction Pattern of Pure Iron is influenced by various thermo-mechanical processing. The present article aims to index new peaks formed in XRD pattern of pure iron when it is electro-polished after rough and fine polishing.

Keywords: Electro-Polishing, thermo-mechanical processing, pure iron

INTRODUCTION:

XRD characterization techniques enable us to study the changes which afflict on the pure iron sample subjected to cold rolling & annealing and other thermo-mechanical processing. By studying the variation in XRD pattern one can roughly estimate the under-lying phenomenon which causes the change in XRD pattern.

Experimental Procedure:

Pure Iron buttons are Melted by Vaccum Induction Melting in protective atmosphere of Argon gas. The resultant Iron conglomerate is characterized for, XRD.

SAMPLE 'S with corresponding weights:



- 1) 23.849
- 2) 23.642
- 3) 22.453
- 4) 21.52
- 5) 25.835 gms

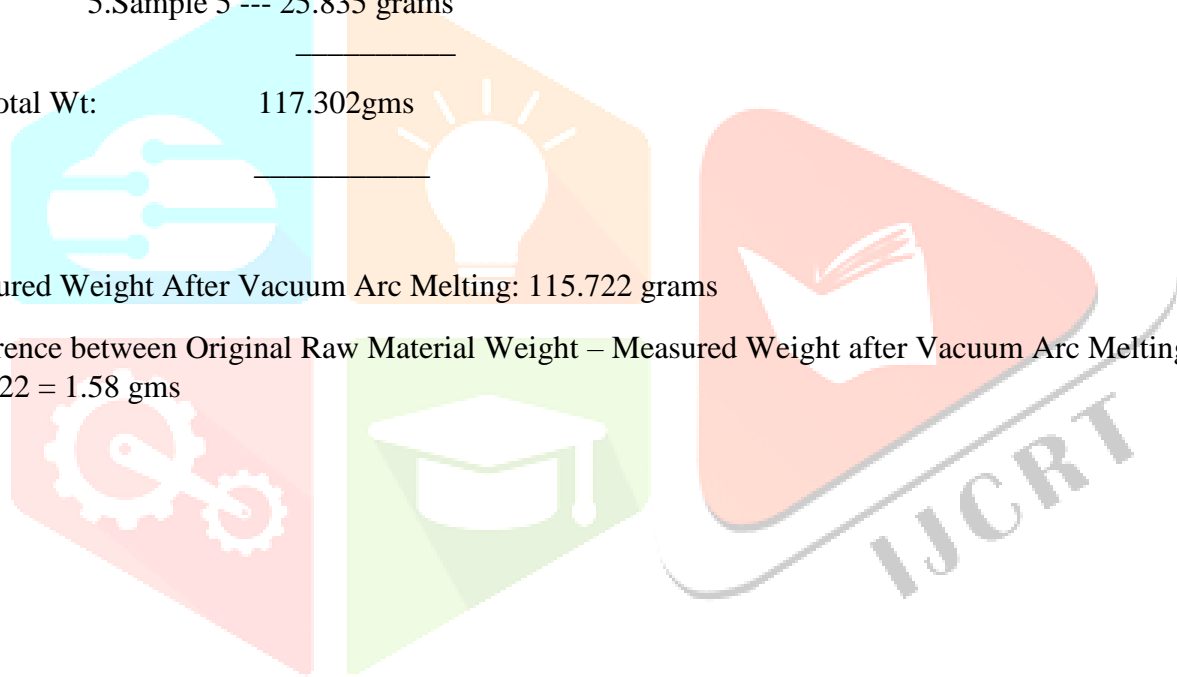
The Samples are melted using Vaccum Arc Melting. Intially first three samples were melted and solidified. Later next two samples were melted with former solidified product to form final product.

| | | | | |
|------------------------------|---|-----|--------|-------|
| 1.Sample | 1 | --- | 23.849 | grams |
| 2. Sample | 2 | --- | 23.642 | grams |
| 3. Sample | 3 | --- | 22.453 | grams |
| 4. Sample 4 --- 21.523 grams | | | | |
| 5.Sample 5 --- 25.835 grams | | | | |

Total Wt: 117.302gms

Measured Weight After Vacuum Arc Melting: 115.722 grams

Difference between Original Raw Material Weight – Measured Weight after Vacuum Arc Melting: 117.302 - 115.722 = 1.58 gms



Thickness of Final Iron Conglomerate: 6mm.

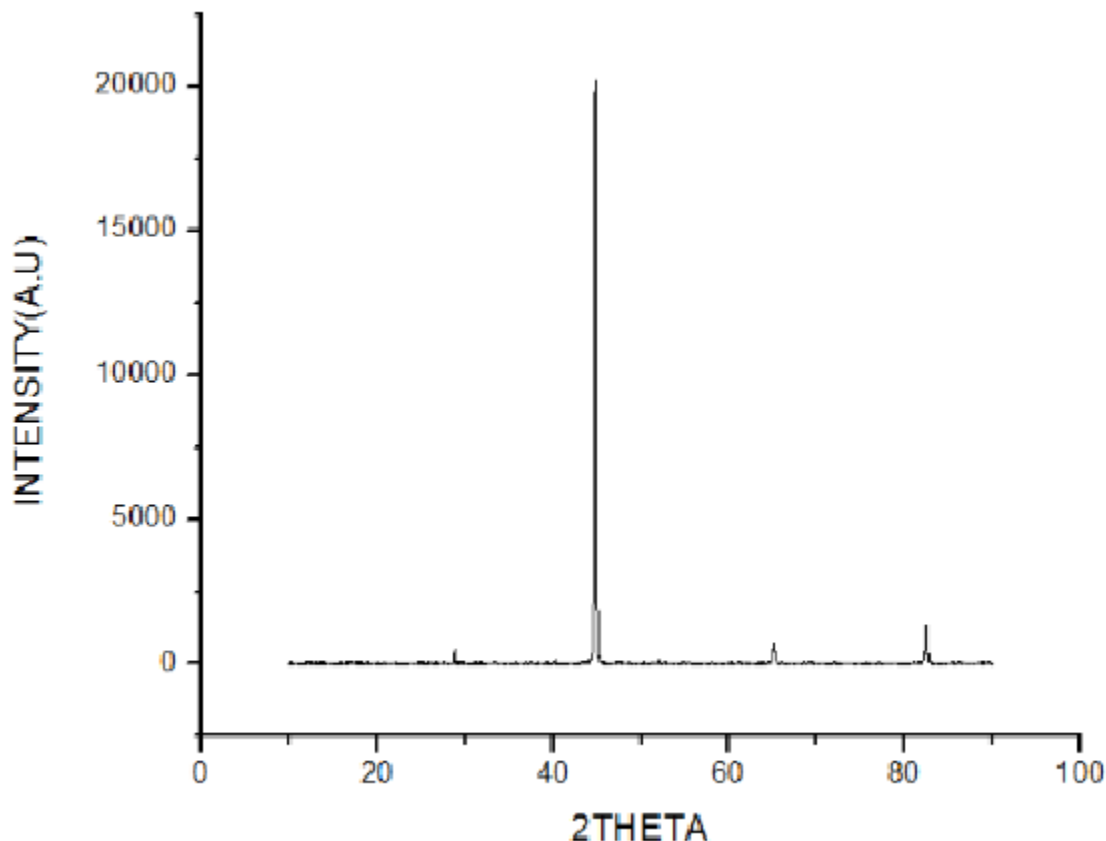


| SAMPLE | SAMPLE 1 | SAMPLE 2 | SAMPLE 3 |
|---------------|----------|----------|----------|
| WEIGHT(gms) | 42.115 | 48.581 | 40.198 |
| THICKNESS(mm) | 7.01 | 7.28 | 6.93 |
| LENGTH(mm) | 31.56 | 32.37 | 32.28 |
| BREADTH | 27.10 | 30.57 | 23.66 |

RESULTS:

XRD pattern of above Sample is Rough and Fine polished and finally Electro-Polished with solution of per chloric acid and ethanol (=1:9),with current 2.26 A,Voltage 35V,time 5 sec,Temperature 10⁰C.

XRD pattern is identified by using TRADITIONAL X RAY DIFFRACTOMETER.



| 2THETA(2θ) | THETA(θ) | SIN(θ) | SIN²θ | RATIOS | [HKL] PLANE | INTER-PLANAR SPACING(d) | LATTICE PARAMETER(a) |
|------------|----------|---------|---------|--------|-------------|-------------------------|----------------------|
| 44.7855 | 22.39275 | 0.3809 | 0.14512 | 1 | 2[110] | 2.0215 | 2.858 |
| 65.1439 | 32.57195 | 0.53835 | 0.2897 | 1.996 | 4[200] | 1.4302 | 2.8604 |
| 82.4486 | 41.2243 | 0.6590 | 0.43428 | 2.992 | 6[211] | 1.1684 | 3.3047 |
| 28.7728 | 14.3864 | 0.24845 | 0.06172 | | | 3.099 | |

Additional peak is noticed at 2THETA: 28.7728[last 2theta(2θ) value]

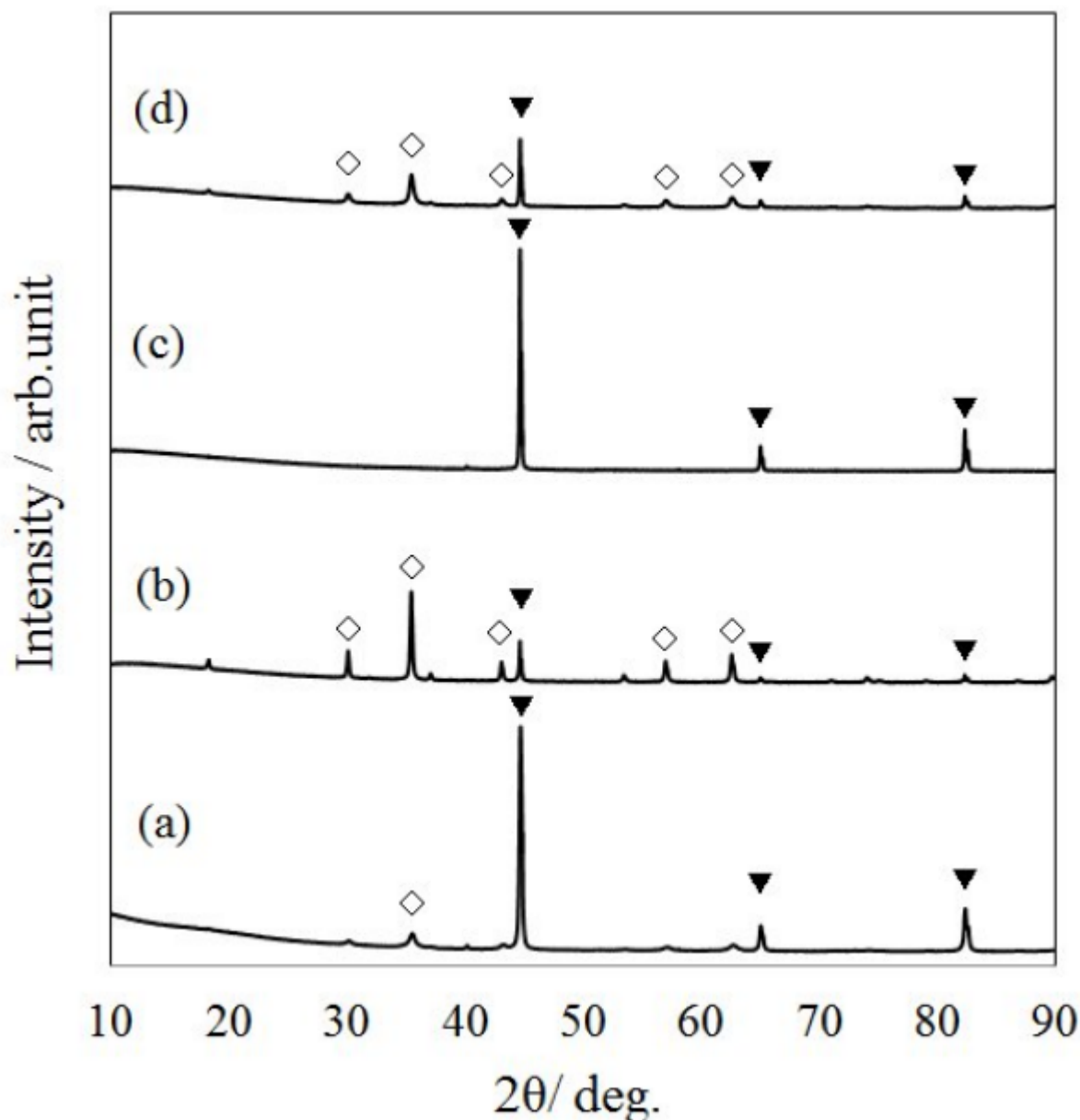
Discussion:

The additional Peak might be because of the diffraction from [410] planes in Fe crystal.

- ⇒ 2THETA= 28.772
- ⇒ THETA(θ)= 14.386
- ⇒ SIN(θ)=0.248453
- ⇒ WAVE LENGTH=0.54A⁰
- ⇒ INTER-PLANAR Spacing = λ/[2*SIN(θ)]
- ⇒ d = 0.54/[2*0.248453]
- ⇒ d = 3.09917 A⁰
- ⇒ LATTICE PARAMETER, a for IRON = 2.866 A⁰
- ⇒ d = a/[√h²+k²+l²]
- ⇒ [√h²+k²+l²] = a /d
- ⇒ h²+k²+l² = (a/d)²

- ⇒ $a = 2.866 \text{ \AA}$, $d = 3.09917 \text{ \AA}$
- ⇒ $h^2 + k^2 + l^2 = 0.85 = [4/5 \ 1/20 \ 0] = [16 \ 1 \ 0]$
- ⇒ $[h \ k \ l] = [4 \ 1 \ 0]$ PLANE

The additional peak formation may be attributed to Fe₃O₄ formation on the crystal as comparisons with XRD of the fresh iron metal.



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CONCLUSIONS: An Additional Peak is registered in XRD pattern of Electro- Polished Iron Sample , diffracting plane is assumed to be [410] planes corresponding indexed Fe₃O₄ compound.

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