# MIDHANI INTERNSHIP WORK

**WORK**

1. Polishing:
   * 1. Paper polishing of grid size – 100, 320, 600, 800, 1000.
     2. Cloth polishing
2. Electropolishing:
   * 1. Aim – To obtain optimising parameters of different steel grades.
     2. Sample of grades – MDN 132 low alloy steel (nitriding steel), 99A low alloy steel, 12X (Low alloy steel)
     3. Solution – A2 solution (20% of perchloric acid and 80% of methanol)
     4. Electrode –
3. EBSD:

**WORK DATA**

**MDN132:**

* 1st we did electro polishing of sample MDN132 using 100ml solution and varying voltage and time, then we obtained following data:

|  |  |  |
| --- | --- | --- |
| **Voltage(V)** | **Time(sec)** | **Observation** |
| 5 | 10 | Pits are formed |
| 2.3 | 10 | Pits are formed |
| 3 | 5 | Not etched |
| 3 | 10 | Not properly done the electropolishing |
| 3.9 | 5 | Pits are formed |

* 2nd time doing the experiment with same solution and varying voltage and time, we obtained following data:

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Voltage(V)** | **Current(A)** | **Time(sec)** | **Temperature(°C)** | **Remarks** |
| 2.5 | 0.1 | 15 | 0 – 10 | Microstructure is not appeared and pits are formed |
| 3 | 0.1 | 15 | 0 – 10 | Pits are formed |
| 4 | 0.0 | 20 | 0 – 10 | Pits are formed |
| 5 | 0.0 | 20 | 0 – 10 | Pits are formed |
| 5 | 0.2 | 10 | 0 - 10 | Pits are formed |

* We did some mistakes which are doing polishing by applying high force on the sample. we should not apply high force on the sample for polishing, because, it will develop stresses in sample the sample. Stresses are developed due to the development of dislocation in the, it will affect the image formation in the EBSD analysis.

**99A:**

* We did electro polishing of the 99A grade steel sample by using the 200ml of solution with same composition. We obtained following data:

|  |  |  |
| --- | --- | --- |
| **Voltage(V)** | **Time(sec)** | **Current(A)** |
| 2 | 10 | 0.1 |
| 1.5 | 10 | 0.1 |
| 1 | 10 | 0 |

* After doing electro polishing at 1V we observed less pits then, we went to do EBSD analysis. In EBSD image is not formed surface smoothness is not enough for EBSD, then we went back to polishing the sample and again we have to electro polishing.
* NOTE:

1. sample must be done dry polishing in cloth polishing.
2. Water act as lubricant in the cloth polishing and reduces the strains developed in the surface.
3. For EBSD analysis strains should not be present because, this strain develop dislocations in surface, this dislocations effect the scattering of beam.

* Again, electro polishing in this case we used liquid N2 + Methanol solution to obtain negative temperature, by placing the electrolyte solution beaker in a beaker containing liquid N2 + Methanol solution to obtain negative temperature in the solution.

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **case** | **round** | **Voltage(V)** | **Time(sec)** | **Current(A)** | **Temperature(°C)** | **Remarks** |
| 1 | 1st round  2nd round | 3.5  3.5 | 15  25 | 0.0  0.0 | <0  <0 | Pits are formed |
| 2 | Only one round | 2.5 | 25 | 0.0 | <0 | Pits are formed |
| 3 | 1st round  2nd round | 2.5  2.5 | 5  5 | 0.6  0.3 | <0  <0 | Microstructure is appeared, pits are formed and over etched |

* We went to EBSD analysis, image is not formed.

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Case** | **Round** | **Voltage(V)** | **Current(A)** | **Time(s)** | **Temp(C)** | **Remarks** |
| 1 | 1st round  2nd round  3rd round | 1.5  2  2.5 | 0  0  0 | 20  10  20 | <0  <0  <0 | Pits and scratches are observed |
| 2 | 1st round | 2 | 0 | 20 | <0 | Light etching but pits are reduced |
| 3 | 1st round | 2.5 | 0 | 20 | <0 | Pits are reduced but scratches are formed |
| 4 | 1st round | 3 | 0 | 20 | <0 | Pits are increased but microstructure is lightly highlighted in the background |

* Went to check EBSD pattern , but pattern is not achieved properly.

**12X:**

* We did electro polishing of the 12X grade steel sample by using the 200ml of solution with same composition liquid N2 + Methanol solution. We obtained following data:

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **case** | **round** | **Voltage(V)** | **Time(sec)** | **Current(A)** | **Temperature(°C)** | **Remarks** |
| 1 | 1st round  2nd round | 4.5  4.5 | 10  20 | 0.0  0.0 | <0  <0 | Less pits |
| 2 | 1st round  2nd round  3rd round | 4.5  4.5  4.5 | 20  5  5 | 0.0  1.3  0.8 | <0 | Microstructure is appeared and no pits. |

**30X series (Low Alloy steel):**

* We did electro polishing of the 30X grade steel sample by using the 200ml of solution with same composition liquid N2 + Methanol solution. We obtained following data:

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **case** | **round** | **Voltage(V)** | **Time(sec)** | **Current(A)** | **Temperature(°C)** | **Remarks** |
| 1 | 1st round  2nd round | 3  3 | 10  20 | 0.2  0.1 | <0  <0 | Less pits  Less pits, but dark spots in some regions |

* We For 3V, 20sec case, we had dark spots it might have been due to over etching, so we go for fine cloth polishing for about a minute to remove the oxide layer formed.
* Yet, after doing all this, we did not get the EBSD pattern for both the cases.

**EBSD SAMPLE PREPARATION:**

* **POLISHING:** Polishing removes the damage created by grinding and prepares the sample for optical, hardness, SEM or EBSD analysis.
* **FACTORS AFFECTING THE SURFACE FINSIH:**
* Abrasive size and type
* Cloth texture
* Polish time
* Specimen load
* Rotational speed
* Rotation direction
* **POLISHING CLOTHS:**

|  |  |
| --- | --- |
| **NAPLESS CLOTH** | **NAPPED CLOTH** |
| Hard | Soft |
| With these clothes, abrasive sits on the surface of the cloth for aggressive polishing | Abrasive penetrates the cloth, allowing less aggressive polishing |
| **Best for maximizing flatness** | **Best for high quality surface finish.** |

* **DIAMOND ABRASIVES:** Mostly used for preparation of most materials due to it’s high material removal rates.

|  |  |
| --- | --- |
| **SUSPENSIONS** | **PASTE** |
| High concentration diamond uniformly suspended, applied using spray bottle or through dispersing system. | Materials such as very soft alloys, pure metals, or refractories are prone to diamond embedding, so paste is better suited for polishing. |

|  |  |
| --- | --- |
| **MONOCRYSTALLINE** | **POLYCRYSTALLINE** |
| Cost effective | Faster prep times |
| Sharp, blocky particles | Reduced deformation, multi-faceted shape |
| **Best for ceramics** | **Only small amount required for high quality surface finish.** |

* **FINAL POLISHING SUSPENSIONS:** They are designed to remove final layer of surface deformation often invisible to the naked eye. Two types of final polishing suspensions are Alumina and Colloidal Silica.

We can see their differences in the table below:

|  |  |
| --- | --- |
| **ALUMINA** | **COLLOIDAL SILICA** |
| Contains seeded gel Alumina, provides effective material removal, with a superior surface finish, via a purely mechanical abrasive process. | Has soft reaction layer to attack the specimen surface. Spherical shape of silica allows it to wipe away top surface layer without scratches. |
| Preferred for Iron, Steel, Stainless steels, copper, polymer, microelectronics and precious metals. | Preferred for Aluminium, refractories, ceramic’s |
| Mechanical polish keeps sample flat, but not completely damage free. | Chemo-mechanical polish creates etching and slight polishing relief, but provides damage free polish. |
| **Best for coating measures and boundary layer analysis.** | **Best for microstructural analysis and EBSD.** |

* Move to next step of polishing only when all the scratches are uniform and evidence of previous step is gone.
* Too much of abrasive can cause hydroplaning with little to no material removal and causes wastage of abrasive.
* Too little of abrasive would cause heat damage.
* To avoid cross contamination, each polishing cloth should only be used for one abrasive size. Applying multiple abrasives on a single cloth can cause scratches on specimen.
* **Relief**: Demonstrated by harder phases being left raised above the surface of softer matrix. Prevent relief by reducing polish time, using a shorter napped cloth, applying diamond paste rather than suspension.
* **Diamond Embedding**: Occurs when harder particles become engrained in softer matrix. Using a fixed abrasive like paste or ultrasonic cleaning between stages can reduce embedding.
* **Smearing**: A superficial but significant form of damage that makes microstructural details less distinct, often caused by soft materials or poor lubrication.
* **Comet tails**: Result of poorly bonded hard phases in softer matrix, pores in matrix result in unidirectional grooves emanating from particles or holes. To avoid this, use hard napless clothes.
* **Smearing:** A Superficial but signficant