X-Ray Diffraction

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Abstract

This experiment utilized X-ray Diffraction (XRD) to analyze the structural properties of various polycrystalline materials including copper (Cu), silicon (Si), sodium chloride (NaCl), and others. Through the application of XRD, we were able to determine the phase compositions and lattice parameters. The results confirmed the crystalline structures and phase purity of Cu and NaCl, showcasing their expected cubic structures and polycrystalline nature. However, discrepancies in the characterization of TiO2 highlighted potential anomalies or procedural errors, suggesting areas for further investigation.

Keywords: *X-ray Diffraction, Crystalline, Phase, Non-destructive*

1 Introduction

XRD is a fundamental analytical method used to investigate the atomic and molecular structure of a crystal. The principle behind XRD is based on the constructive interference of monochromatic X-rays and a crystalline sample. This experiment aims to employ XRD to explore the structural properties of polycrystalline materials, identify crystalline phases

When X-rays interact with a crystalline material, they are diffracted in many specific directions. According to Bragg's Law ($n\lambda = 2d\sin\theta$), these directions depend on the wavelength (λ) of the X-rays and the distance between the lattice planes in the crystal (d). Here, n represents an integer, and θ is the angle of incidence that satisfies the condition for constructive interference. This law facilitates the prediction of where diffracted peaks will appear in XRD patterns, allowing for accurate identification of the material's phase based on its unique crystal structure.

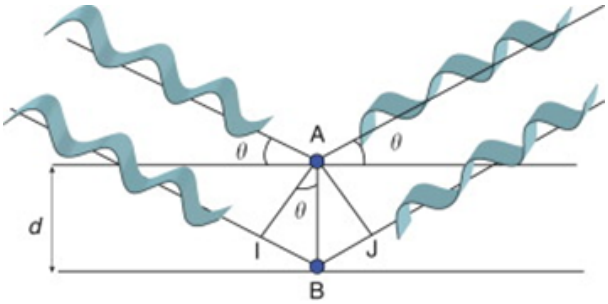

Figure 1: Bragg's Law

The experiment will utilize a diffractometer equipped with an X-ray tube, a sample holder, and a detector, configured to measure the angle (θ) and intensity of the diffracted beams. As the sample is rotated, the detector records the diffracted X-rays at various angles, creating a diffractogram that reveals information about the crystal structure. Analyzing these patterns enables the determination of the lattice parameters, providing insights into the material's crystalline structure.

2 Experimental Details

This section details the procedure for the experiment and the expected results according to the lab manuals and the relevant equations.

1. **Equipment and Materials:**
    - Panalytical Xpert Pro 3 powder diffractometer for high-resolution XRD analysis.
    - Polycrystalline metallic sheets and glass microscope slides as samples.
    - Personal Protective Equipment (PPE): Laboratory coats, gloves, and eye protection.
2. **Procedure:**
    (a) **Setup:**
        - Ensure all diffractometers are properly set up and functioning. Check power, generator settings, alignment of the X-ray tube, and detectors.
        - Properly mount the sample in the holder of the Xpert Pro 3 diffractometer, ensuring correct orientation for diffraction.

    (b) **XRD Measurement:**
        - Configure the Xpert Pro 3 to use Cu Kα radiation (wavelength = 1.5418 Å). Set the operational voltage and current (typically 40kV and 30mA).
        - Initiate the scan over a 2θ range of about 10° to 100° to capture all relevant diffraction peaks.

    (c) **Data Collection:**
        - Record the intensity of the diffracted X-rays at each angle of 2θ. Monitor equipment continuously to adjust for any drift.

    (d) **Data Analysis:**
        - Apply Bragg's Law to determine the d-spacings and identify the phases using standard reference patterns. This is done through HighScore.

    (e) **Safety Measures:**
        - Ensure the X-ray generator is off when not in use and during sample loading/unloading.
        - Wear appropriate PPE throughout the experiment to mitigate exposure risks.

3 Results and Discussion

The following sections contain our results for each method that includes tables and graphs. This is accompanied by a discussion that includes interpretations of the results. We conducted XRD on 5 samples: Copper (Cu), Halite composed of Sodium Chloride (NaCl), Silicon (Si), Strontianite composed of Strontium Carbonate ($SrCO_3$), Rutile composed of Titanium(II) dioxide (TiO_2). The data generated by HighScore was inconsistent as the documents did not all contain the XRD spectrum. Therefore using the csv/xrdml file for each sample I plotted the graph on RStudio. For each sample I include the peaks list with it, longer lists are in the appendix to maintain the flow of the report. The calculated lattice parameters by HighScore are in the appendix, note that Silicon did not generate a HighScore document.

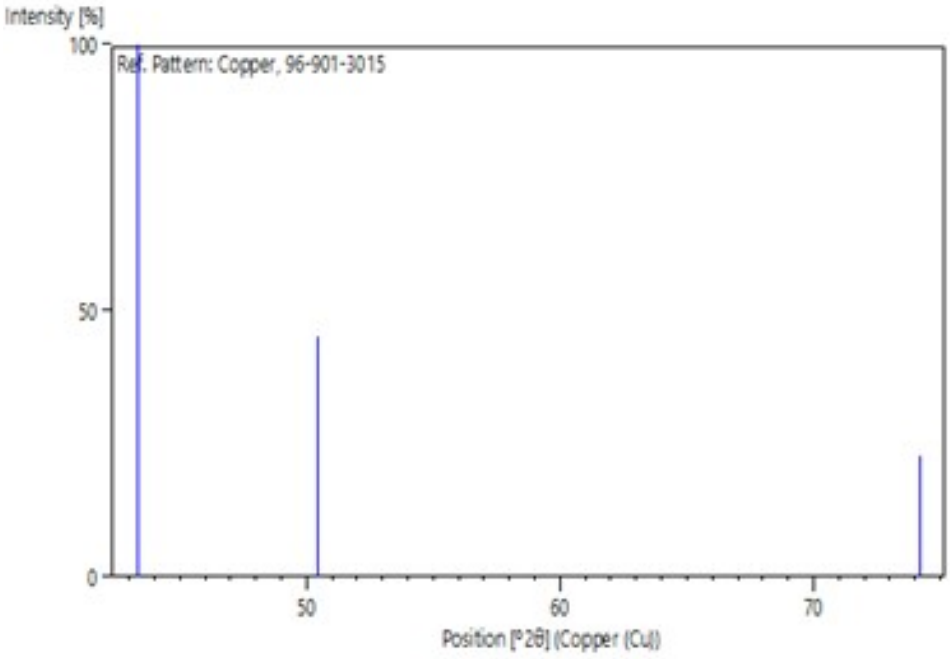

Figure 2: Cu XRD Spectrum

No.	h	k	l	d (Å)	2θ (°C)	I (%)
1	1	1	1	2.086	43.34	100
2	0	2	0	1.8065	50.48	45.3
3	0	2	2	1.2774	74.17	22.7

Table 1: Cu Peaks List

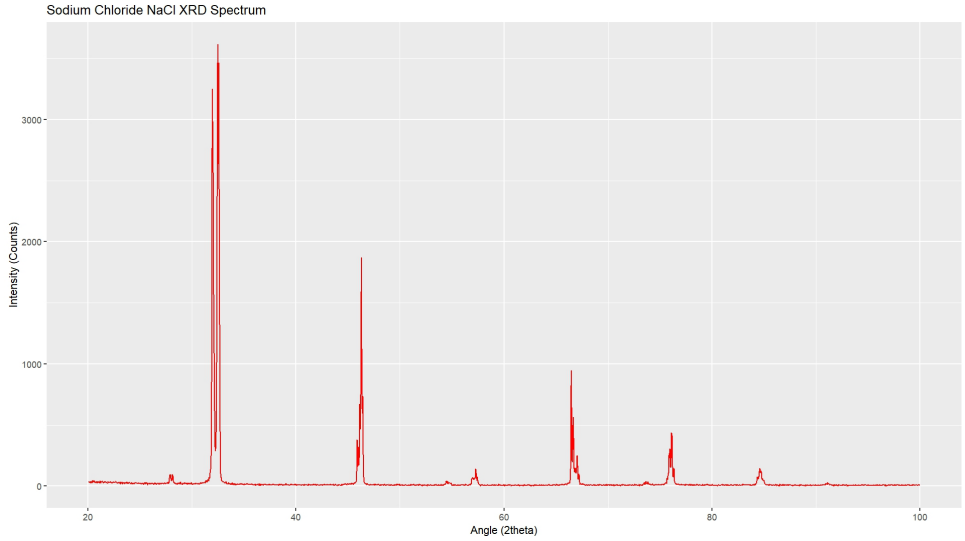

Figure 3: NaCl XRD Spectrum

Table 2: NaCl Peaks List

No.	h	k	l	d (Å)	2θ (°C)	I (%)
1	1	1	1	3.2326	27.57	8.7
2	0	2	0	2.7995	31.94	100
3	0	2	2	1.9796	45.8	64.1
4	1	3	1	1.6882	54.3	2.2
5	2	2	2	1.6163	56.93	20.1
6	0	4	0	1.3998	66.78	8.6
7	1	3	3	1.2845	73.7	1
8	0	4	2	1.252	75.94	22.4
9	2	4	2	1.1429	84.75	16.2

For both Cu and NaCl, the experiment corresponds greatly to the sources, indicating the accuracy of the experimental method. Agreeing with both samples being polycrystalline and having a cubic structure

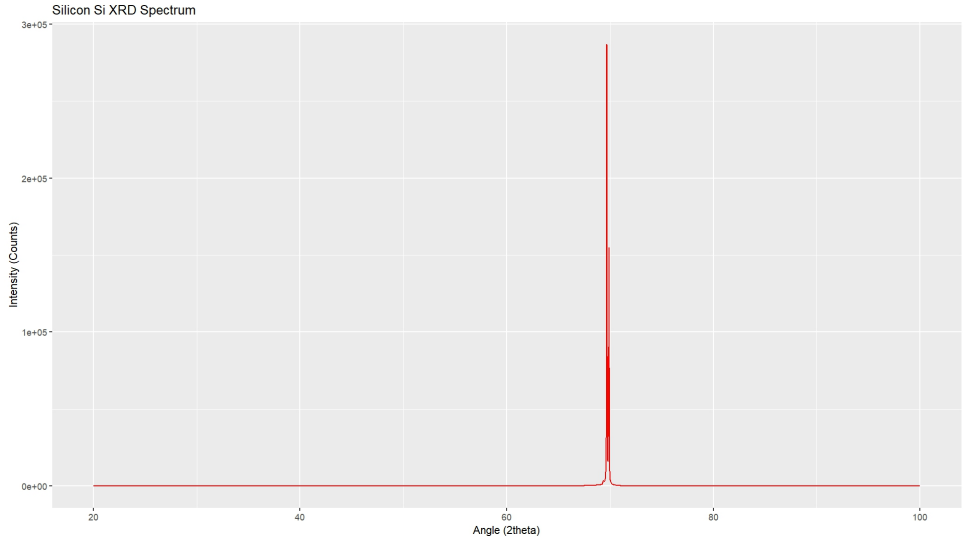

Figure 4: Si XRD Spectrum

that the PS layer is still crystalline in this illustration. Other researchers [12] have come to the same conclusion.

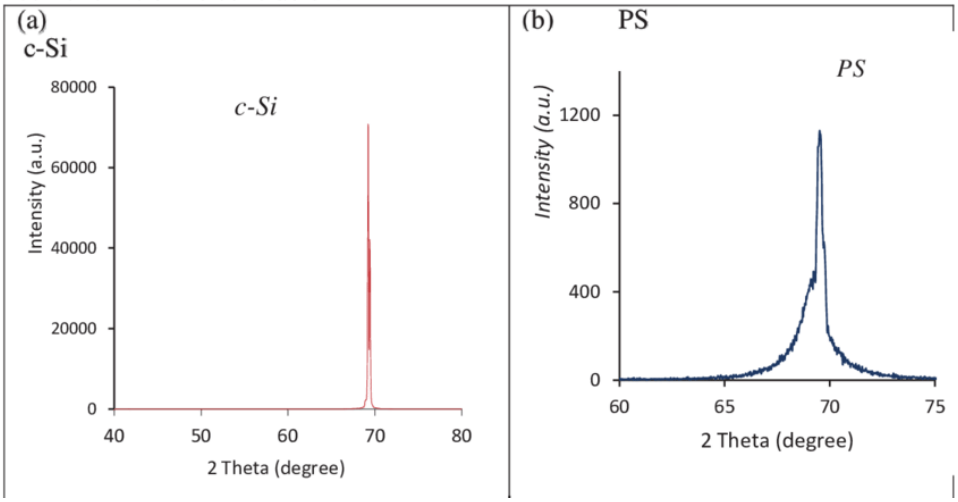

Figure 5: Si XRD Spectrum Reference

The HighScore word document for Silicon was not generated, therefore browsing online, an XRD pattern for a p-type silicon crystalline silicon corresponds to our XRD pattern. Left image is the p-type and right image is a porous silicon.

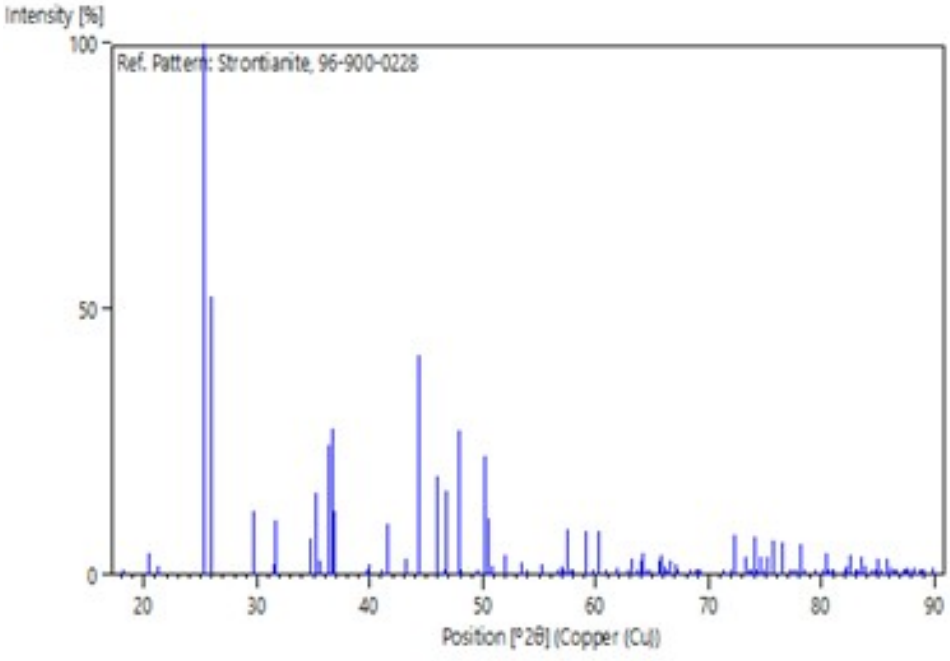

Figure 6: $SrCO_3$ XRD Spectrum

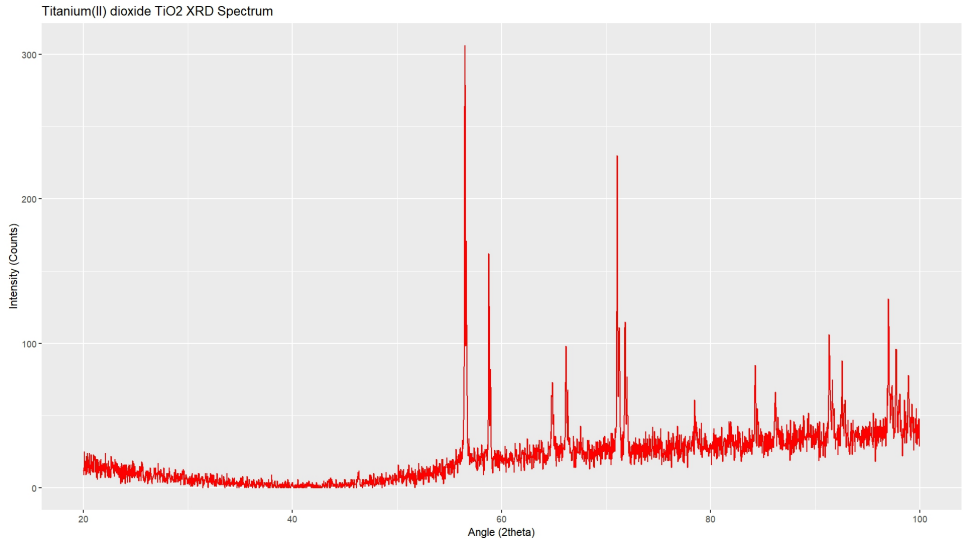

Figure 7: TiO_2 XRD Spectrum

The peaks list for $SrCO_3$ and TiO_2 are in the appendix. Comparing to the sources, $SrCO_3$ prominent peaks are reflected in the source. While for TiO_2 it is not reflected in the source, suggesting a mischaracterization of the sample, or improper procedure. The noise is expected as Anatase and Rutile phases coexist in the sample.

4 Applications

- Qualitative and quantitative phase analysis of pure substances and mixtures.
- Analysis of phase changes under non-ambient conditions like temperature and pressure.
- Analysis of physical properties such as crystallite size, crystal orientation, and residual stress.
- Use in polycrystalline layered materials such as coatings and thin films via grazing incidence XRD (GIXRD).
- Microdiffraction studies for examining small areas within polycrystalline materials.
- High-resolution analysis of single crystal semiconductor wafers or epitaxial layers using HR-XRD.
- Examination of non-crystalline components of materials using various X-ray scattering methods.

5 Conclusion

The experiment successfully demonstrated the application of XRD in identifying and characterizing the crystalline structures of different materials. The results closely matched the expected outcomes based on standard references, indicating the reliability of the experimental setup and procedure. For materials like Cu and NaCl, the XRD patterns confirmed their polycrystalline nature and cubic structures, aligning with theoretical predictions and literature values. However, discrepancies in the characterization of TiO2 suggest potential experimental errors or sample anomalies. This experiment underscores the efficacy of XRD in material science, particularly in validating material properties and improving the understanding of material behaviors under standard environmental conditions.

6 Appendix

Table 3: Cu HighScore Information

Name and formula	
Reference code:	96-901-3015
Mineral name:	Copper
Compound name:	Copper
Common name:	Copper
Chemical formula:	Cu4.00
Crystallographic parameters	
Crystal system:	Cubic
Space group:	F m -3 m
Space group number:	225
a (Å):	3.613
b (Å):	3.613
c (Å):	3.613
Alpha (°):	90
Beta (°):	90
Gamma (°):	90
Calculated density (g/cm3):	8.95
Volume of cell (e6 pm3):	47.16
RIR:	9.63
Subfiles and quality	
Subfiles:	User Inorganic, User Metallic, User Mineral
Quality:	User From Structure (=)
Comments	
Creation Date:	6/15/2016 8:20:51 PM
Modification Date:	6/15/2016 8:20:51 PM
Cross-References:	ICDD:96-901-3015
Publication title:	High-temperature thermal expansion of six metallic elements measured by dilatation method and X-ray diffraction Locality: synthetic Sample: at $T = 293K$
COD database code: 9013014	

Structure								
No.	Name	Element	X	Y	Z	Biso	sof	Wyck.
1	Cu	Cu	0	0	0	0.5	1	4a

Table 4: NaCl HighScore Information

Name and formula	
Reference code:	96-900-6371
Mineral name:	Halite
Compound name:	Halite
Common name:	Halite
Chemical formula:	Na4.00Cl4.00
Crystallographic parameters	
Crystal system:	Cubic
Space group:	F m -3 m
Space group number:	225
a (Å):	5.599
b (Å):	5.599
c (Å):	5.599
Alpha (°):	90
Beta (°):	90
Gamma (°):	90
Calculated density (g/cm3):	2.21
Volume of cell (e6 pm3):	175.52
RIR:	4.98
Subfiles and quality	
Subfiles:	User Inorganic, User Mineral
Quality:	User From Structure (=)
Comments	
Creation Date:	6/15/2016 8:10:15 PM
Modification Date:	6/15/2016 8:10:15 PM
Cross-References:	ICDD:96-900-6371
Publication title:	Thermal expansion of alkali halides at high pressure: NaCl as an example Sample: T = 100 K, Molar volume = 26.42 cc/mol
COD database code:	9006370

Structure

No.	Name	Element	X	Y	Z	Biso	sof	Wyck.
1	Na	Na	0	0	0	N/A	N/A	N/A
2	Cl	Cl	0.5	0.5	0.5	N/A	N/A	N/A

Table 5: SrCO3 HighScore Information

Name and formula	
Reference code:	96-900-0228
Mineral name:	Strontianite
Compound name:	Strontianite
Common name:	Strontianite
Chemical formula:	Sr4.00C4.00O12.00
Crystallographic parameters	
Crystal system:	Orthorhombic
Space group:	P n m a
Space group number:	62
a (Å):	5.997
b (Å):	5.09
c (Å):	8.358
Alpha (°):	90
Beta (°):	90
Gamma (°):	90
Calculated density (g/cm3):	3.84
Volume of cell (e6 pm3):	255.13
RIR:	3.97
Subfiles and quality	
Subfiles:	User Inorganic, User Mineral
Quality:	User From Structure (=)
Comments	
Creation Date:	6/15/2016 7:59:46 PM
Modification Date:	6/15/2016 7:59:46 PM
Cross-References:	ICDD:96-900-0228
Structure TIDY:	TRANS c,a,b origin 0 1/2 0
Structure TIDY:	REMARK Transformed from setting P m c n.
Publication title:	Crystal structures of aragonite, strontianite, and witherite
COD database code: 9000227	

Structure								
No.	Name	Element	X	Y	Z	Biso	sof	Wyck.
1	Sr	Sr	0.2431	0.25	0.584	0.5077	1	4c
2	C	C	0.0864	0.25	0.2399	0.5922	1	4c
3	O1	O	0.0946	0.25	0.0881	0.9127	1	4c
4	O2	O	0.0839	0.0306	0.3179	0.7967	1	8d

Table 6: TiO2 HighScore Information

Name and formula	
Reference code:	96-231-0487
Compound name:	Ti O2
Common name:	Ti O2
Chemical formula:	O8.00Ti4.00
Crystallographic parameters	
Crystal system:	Orthorhombic
Space group:	P b c n
Space group number:	60
a (Å):	4.515
b (Å):	5.497
c (Å):	4.939
Alpha (°):	90
Beta (°):	90
Gamma (°):	90
Calculated density (g/cm3):	4.33
Volume of cell (e6 pm3):	122.58
RIR:	3.1
Subfiles and quality	
Subfiles:	User Inorganic
Quality:	User From Structure (=)
Comments	
Creation Date:	6/14/2016 7:23:43 PM
Modification Date:	6/14/2016 7:23:43 PM
Cross-References:	ICDD:96-231-0487
Structure TIDY:	TRANS Origin 1/2 0 1/2
Publication title:	The structure of Ti O2 II, a high-pressure phase of Ti O2
COD database code: 2310486	

Structure								
No.	Name	Element	X	Y	Z	Biso	sof	Wyck.
1	O1	O	0.286	0.124	0.088	0	1	8d
2	Ti1	Ti	0	0.329	0.25	0	1	4c

Table 7: SrCO3 Peaks List 1

No.	h	k	l	d (Å)	2θ (°C)	I (%)
1	1	0	1	4.8725	18.192	0.2
2	0	1	1	4.34729	20.412	4.3
3	0	0	2	4.179	21.244	1.8
4	1	1	1	3.51976	25.283	100
5	1	0	2	3.42864	25.966	52.7
6	2	0	0	2.9985	29.772	12
7	1	1	2	2.84366	31.434	2
8	2	0	1	2.82237	31.677	10.3
9	2	1	0	2.58354	34.694	6.7
10	0	2	0	2.545	35.236	15.7
11	1	0	3	2.52666	35.501	2.6
12	2	1	1	2.46831	36.369	24.5
13	0	1	3	2.44387	36.745	27.5
14	2	0	2	2.43625	36.864	12.2
15	1	1	3	2.26316	39.798	0
16	1	2	1	2.25582	39.933	2.2
17	2	1	2	2.19751	41.04	0
18	0	2	2	2.17364	41.511	9.8
19	0	0	4	2.0895	43.265	3
20	1	2	2	2.04355	44.289	41.2
21	2	0	3	2.04099	44.347	0.2
22	1	0	4	1.97316	45.957	18.6
23	3	0	1	1.94417	46.683	0.1
24	2	2	0	1.94032	46.781	16
25	2	1	3	1.89437	47.986	27.3
26	2	2	1	1.89006	48.102	0.4
27	1	1	4	1.83976	49.504	0.8
28	3	1	1	1.81619	50.191	22.4
29	3	0	2	1.80331	50.575	10.5
30	1	2	3	1.79307	50.884	1.8
31	2	2	2	1.75988	51.914	3.6
32	2	0	4	1.71432	53.402	2.4
33	3	1	2	1.69978	53.895	0.4
34	0	3	1	1.66275	55.196	2
35	2	1	4	1.62465	56.606	0
36	3	0	3	1.62417	56.624	0
37	0	2	4	1.61493	56.977	1.8
38	1	0	5	1.61022	57.16	0.6
39	1	3	1	1.6023	57.468	8.6
40	2	2	3	1.59222	57.866	0
41	0	1	5	1.58815	58.029	1
42	1	2	4	1.55938	59.205	8.2
43	3	1	3	1.5473	59.713	0
44	3	2	1	1.54495	59.814	0.1
45	1	1	5	1.53523	60.232	8.1

Table 8: SrCO3 Peaks List 2

No.	h	k	l	d (Å)	2θ (°C)	I (%)
46	1	3	2	1.52066	60.869	0.9
47	4	0	0	1.49925	61.833	1.4
48	2	3	0	1.47666	62.886	0.2
49	4	0	1	1.4757	62.932	1.1
50	3	2	2	1.47138	63.138	3
51	2	0	5	1.46005	63.685	0
52	2	3	1	1.45414	63.974	2.7
53	0	3	3	1.4491	64.223	4.3
54	3	0	4	1.44444	64.455	1.2
55	4	1	0	1.43816	64.771	0
56	2	2	4	1.42183	65.608	2.9
57	4	1	1	1.41733	65.842	3.8
58	4	0	2	1.41118	66.166	1.7
59	1	3	3	1.40856	66.305	0.1
60	2	1	5	1.40345	66.578	2.6
61	0	0	6	1.393	67.143	2.2
62	2	3	2	1.3923	67.182	0.1
63	3	1	4	1.38957	67.331	0.2
64	3	2	3	1.36912	68.475	0
65	1	2	5	1.36073	68.956	0.2
66	4	1	2	1.35989	69.005	0.2
67	1	0	6	1.35688	69.18	0
68	4	0	3	1.32023	71.389	0.1
69	1	1	6	1.31109	71.963	0
70	2	3	3	1.30472	72.37	7.6
71	4	2	0	1.29177	73.213	3.3
72	1	3	4	1.28647	73.564	0
73	3	0	5	1.28234	73.84	0
74	3	3	1	1.27833	74.11	7.1
75	4	1	3	1.27794	74.137	7
76	4	2	1	1.27661	74.227	0
77	0	4	0	1.2725	74.507	3.3
78	2	2	5	1.26644	74.925	0.2
79	2	0	6	1.26333	75.141	3.4
80	3	2	4	1.25621	75.641	6.5
81	3	1	5	1.24348	76.555	6.1
82	3	3	2	1.23571	77.125	0.1
83	4	2	2	1.23415	77.24	1
84	1	4	1	1.23121	77.459	0
85	2	1	6	1.22613	77.84	0.3
86	0	2	6	1.22193	78.158	5.7
87	4	0	4	1.21813	78.45	0.7
88	0	4	2	1.21732	78.512	0.3
89	2	3	4	1.20592	79.4	0
90	1	2	6	1.19733	80.084	0

Table 9: TiO_2 Peaks List

No.	h	k	l	d (Å)	2θ (°C)	I (%)
1	1	1	0	3.48898	25.51	45.5
2	1	1	1	2.84967	31.366	100
3	0	2	0	2.7485	32.552	9.7
4	0	0	2	2.4695	36.351	11.7
5	0	2	1	2.40167	37.415	10.3
6	2	0	0	2.2575	39.902	3
7	1	0	2	2.1666	41.652	11
8	1	2	1	2.12035	42.605	18.3
9	1	1	2	2.01568	44.934	9.5
10	2	1	1	1.9234	47.217	1.2
11	0	2	2	1.83694	49.586	5.7
12	2	2	0	1.74449	52.407	4.8
13	1	2	2	1.70151	53.836	0
14	1	3	0	1.69784	53.962	16.9
15	2	0	2	1.66621	55.072	20.5
16	2	2	1	1.6449	55.847	31.8
17	1	3	1	1.60562	57.338	0.4
18	2	1	2	1.59457	57.773	0.6
19	1	1	3	1.4889	62.311	16.5
20	3	1	0	1.45158	64.101	0.4
21	2	2	2	1.42484	65.452	4.4
22	0	2	3	1.41235	66.105	14.1
23	1	3	2	1.39908	66.813	12.7
24	3	1	1	1.39268	67.161	13.9
25	0	4	0	1.37425	68.184	3
26	2	3	1	1.36709	68.59	0.3
27	1	2	3	1.34794	69.705	0
28	0	4	1	1.32395	71.157	4.8
29	2	1	3	1.29287	73.14	0
30	3	0	2	1.28515	73.652	0.7
31	3	2	1	1.27529	74.316	1.3
32	1	4	1	1.27046	74.647	0
33	3	1	2	1.2514	75.984	3.1
34	0	0	4	1.23475	77.196	1
35	2	3	2	1.23274	77.345	0.2
36	0	4	2	1.20083	79.803	0.1
37	2	2	3	1.19733	80.084	1.1
38	1	0	4	1.19102	80.595	0.7
39	1	3	3	1.18192	81.345	0
40	2	4	0	1.17385	82.023	0
41	3	2	2	1.16417	82.855	0
42	1	1	4	1.16401	82.869	1.3
43	3	3	0	1.16299	82.957	2.1
44	1	4	2	1.16049	83.176	1.5
45	2	4	1	1.14204	84.83	5.6
46	3	3	1	1.13203	85.759	0
47	4	0	0	1.12875	86.069	2.9
48	0	2	4	1.12631	86.3	1
49	1	2	4	1.09282	89.638	0

7 References

- Lab manual
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