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Preparation, characterization and magnetic properties for the sample obtained from the polymeric precursor technique

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Preparation of ceramic oxide by polymeric precursor technique is a new advancement in Materials Science. Preparation of $Y_1Ba_2Cu_4O_8$ by polymeric precursor technique has been already reported in the Journal of the Indian Chemical Society. But the sample contains sodium oxide impurity. To control formation of sodium oxide by product, it has been already found that filtration of the precipitate containing mass, obtained during preparation of polyvalent metal ion bound polyacrylate before drying, is necessary. By adopting this modification, an oxide sample has been prepared, in present investigation, which is expected to be antiferromagnetic as is evidenced by ZFC curve. To characterize the sample, XRD, SEM and EDS are done and reported in this present investigation. Magnetic measurements are done to understand antiferromagnetic nature.

Keyword: Ceramic oxide, XRD, Magnetometer, Microscopy, Atiferromagnetic, Polymeric precursor.

1. Introduction

Preparation of Y-Ba-Cu-O by polymeric precursor technique has been already reported [1-3]. It has been found that modified natural polymer, i.e. guargum-graft-acrylamide binds polyvalent metal ions like Y^{3+}, Ba^{2+} and Cu^{2+} in specified conditions [4-6]. It is also reported that --COOH group from the polymer is mainly responsible for binding polyvalent metal ion by guargum-graft-acrylamide [5, 7]. So sodium polyacrylate may be a better choice since it can provide large number of $–COO^{–}$ groups. Preparation of $Y_1Ba_2Cu_4O_8$ by polymeric precursor technique using sodium polyacrylate as the polymeric part has been already reported in the Journal of The Indian Chemical Society [3]. Major problem of this work was formation of sodium oxide by product. It is already reported that if filtration is not done during preparation of polyvalent metal ion bound polyacrylate before drying, NaOH flakes will appear in the precipitate after drying, which will give sodium oxide in the final oxide sample obtained after removing polymeric part by heating at 1000 ^{0}C for 1 hour. So, in this present investigation, filtration has been done before drying. The final oxide sample obtained after removing polymeric part, has been examined by XRD, SEM and EDS, which indicate $Y_1Ba_2Cu_4O_8$ in the sample [3]. In this present investigation, magnetic behavior of the oxide sample obtained has been studied without washing the oxide sample [8] and this work indicates that material is having antiferromagnetic behavior. So the material may be useful. Antiferromagnets can couple to ferromagnets to find use in spin valves which are the basis of magnetic sensors. Fantasy of preparation of Y-Ba-Cu-O by polymeric precursor technique may be the ease of processing of the polymeric precursor, if its rheological properties are known [9].

2. Materials and Brief Methods

2.1 Metal nitrate solutions

$Y(NO_3)_3$ or $Cu(NO_3)_2$ solution is prepared by dissolving Y_2O_3 or CuO in nitric acid to get approximately 0.2(M) solution. $Ba(NO_3)_2$ solution is prepared by dissolving $Ba(NO_3)_2$ in distilled water to get approximately 0.1(M) solution (saturated solution).

2.2 Sodium polyacrylate:
Polyacrylic acid 2100 sodium salt (Fluka) (from Sigma Aldrich) has been used in this work. Material is soluble in water.

2.3 Preparation of polyvalent metal ion bound polyacrylate:
50 ml. approximately 2.9% sodium polyacrylate solution is mixed with 10 ml. $Y(NO_3)_3$ solution, 36 ml. $Ba(NO_3)_2$ solution and 24 ml. $Cu(NO_3)_2$ solution. 20 ml. 60% NaOH solution is added. A bluish precipitate obtained. It is allowed to settle overnight and then liquid is decanted off. Then 45 ml. methyl alcohol is added and allowed to settle and then liquid is decanted off. Process is repeated for several times. Then mixture is filtered and dried in an oven.

2.4 Preparation of the oxide sample
Dried mass is heated to 1000 ^{0}C for one hour to remove polymeric part. The hard solid mass obtained (probably because of sintering) is powered by a mortar and pestle for XRD, SEM, EDS and magnetic measurements.

2.5 XRD
X-ray diffraction (XRD) study has been carried out using powdered sample by using the instrument: D 8 ADVANCE (BRUKER) made in Germany. Copper target (K_α) is used for this experiment.

2.6 SEM and EDS
Scanning Electron Microscopic (SEM) experiment and Energy Dispersive Spectroscopy (EDS) for the sample have been carried out using SCANNING MICROSCOPE (JEOL company made in Japan) model number JSM 5800 using gold coated sample. Gold coating has been done by SPUTTERING TECHNIQUE. EDS study has been carried out using Oxford Instruments (INCA Software). In EDS, peak for gold is ignored.

2.7 Magnetic Measurements
MPMS SQUID VSM (Magnetic Property Measurement System uses Superconducting Quantum Interference Device and Vibrating Sample Magnetometer) has been used to check magnetic properties. 3.65 mg sample is used for magnetic measurements. Sample is cooled in zero field from 380K to 5K. For measurement a field (200 oersted) is imposed to get curve for zero field cooling and curve for field cooling. For zero field cooling curve, temperature is increased from 5K to 380K and for field cooling curve, temperature is decreased from 380K to 5K using a field (200 oersted). In moment vs. field case, ±5 Tesla is considered.

1 Tesla => 10^4 oersted

Moment vs. field plots are considered for set temperature 300K and also for 77K.

3. Results and Discussion
3.1 XRD
XRD pattern for the sample is shown in fig.1. Many peaks match with $Y_1Ba_2Cu_4O_8$. Stick pattern describing matching with peaks for $Y_1Ba_2Cu_4O_8$ is shown in fig.2. XRD study shows strong indication of $Y_1Ba_2Cu_4O_8$ but not of Na_2O and BaO. $Y_1Ba_2Cu_4O_8$ has base centered orthorhombic lattice. $Y_1Ba_2Cu_4O_8$ will contribute in magnetic behavior.

3.2 SEM and EDS
SEM image has been shown in fig.3. SEM image indicated presence of rod-like phase, spherical particle-like phase, white spherical lumps, broken brick block-like phase etc.

Bulk EDS study indicates presence of the following:

Element	Weight%	Atomic%
O K	20.38	42.70
Na K	24.94	36.37
Cu K	21.76	11.48
Y L	10.71	4.04
Ba L	22.21	5.42
Totals	100.00	

Bulk EDS study indicates, still presence of some amount of sodium compound in the oxide sample obtained.

EDS study of the rod-like phase (fig.4) indicates presence of the following:

Element	Weight%	Atomic%
O K	44.48	62.90
Na K	30.30	29.82
Cu K	8.94	3.18
Y L	15.78	4.02
Ba L	0.49	0.08
Totals	100.00	

So this is sodium-rich phase.

EDS study of the spherical particle-like phase (fig.5) indicates presence of the following:

Element	Weight%	Atomic%
O K	24.30	54.82
Na K	10.42	16.36
Cu K	22.04	12.52
Y L	34.53	14.02
Ba L	8.70	2.29
Totals	100.00	

EDS study of the white spherical lumps (fig.6) indicates presence of the following:

Element	Weight%	Atomic%
O K	30.30	61.21
Na K	10.02	14.09
Cu K	23.02	11.71
Y L	34.04	12.37
Ba L	2.61	0.62
Totals	100.00	

EDS study of the broken brick block-like phase (fig.7) indicates presence of the following:

Element	Weight%	Atomic%
O K	19.47	54.23
Na K	10.23	19.82
Cu K	6.28	4.41
Y L	4.37	2.19
Ba L	59.65	19.35
Totals	100.00	

This is barium-rich phase. But it has been found from previous work [10] that BaO formation can be minimized by proper control of addition of amount of $Ba(NO_3)_2$ solution.

SEM and EDS studies indicate sample contains mainly Y-Ba-Cu-O. Although some sodium-rich phase and barium-rich phase are also present.

3.3 Magnetic Measurements

Moment vs. temperature plots are shown in fig. 8. Symbol of magnetic dipole moment in emu, Gau is erg G^{-1} (where G => Gauss).

In fig.8, at near about 325K, there is bifurcation of two curves: curve (i) for zero field cooling and curve (ii) for field cooling. In zero field cooling, when temperature is increased from low temperature, due to freezing of magnetic domains, probably alignment is poorer than that in field cooling case for the same temperature. So moment values for ZFC & FC cases are generally different for the same temperature value. In ZFC curve, there is an interesting feature at very low temperature. When temperature is increased from 5K, moment increases and it reaches to maximum value and then decreases. This is in favour of antiferromagnetic behavior. In FC curve it is not very much obvious; probably because of cooling in presence of field.

At set temperature 300K, moment vs. field plot (fig. 9) is not linear. For paramagnetic substance graph will be linear. But the graph is not linear. For antiferromagnetic substance, saturation is not attained easily. In this case material is showing antiferromagnetic behavior at very low temperature as is evident from M-T plot. M-H plot at 300K is showing hysteresis loop probably because of spin-canting effect in presence of field. But at lower temperature (77K), M-H plot (fig. 10) indicates material is showing differences in magnetic nature than that at 300K in presence of a field. This is probably because of better diamagnetic character of the material at lower temperature than that at 300K in presence of field. This may go in favour of superconductivity at lower temperature [11]. Generally superconducting material becomes diamagnetic at the temperature of showing superconductivity by forming cooper pair.

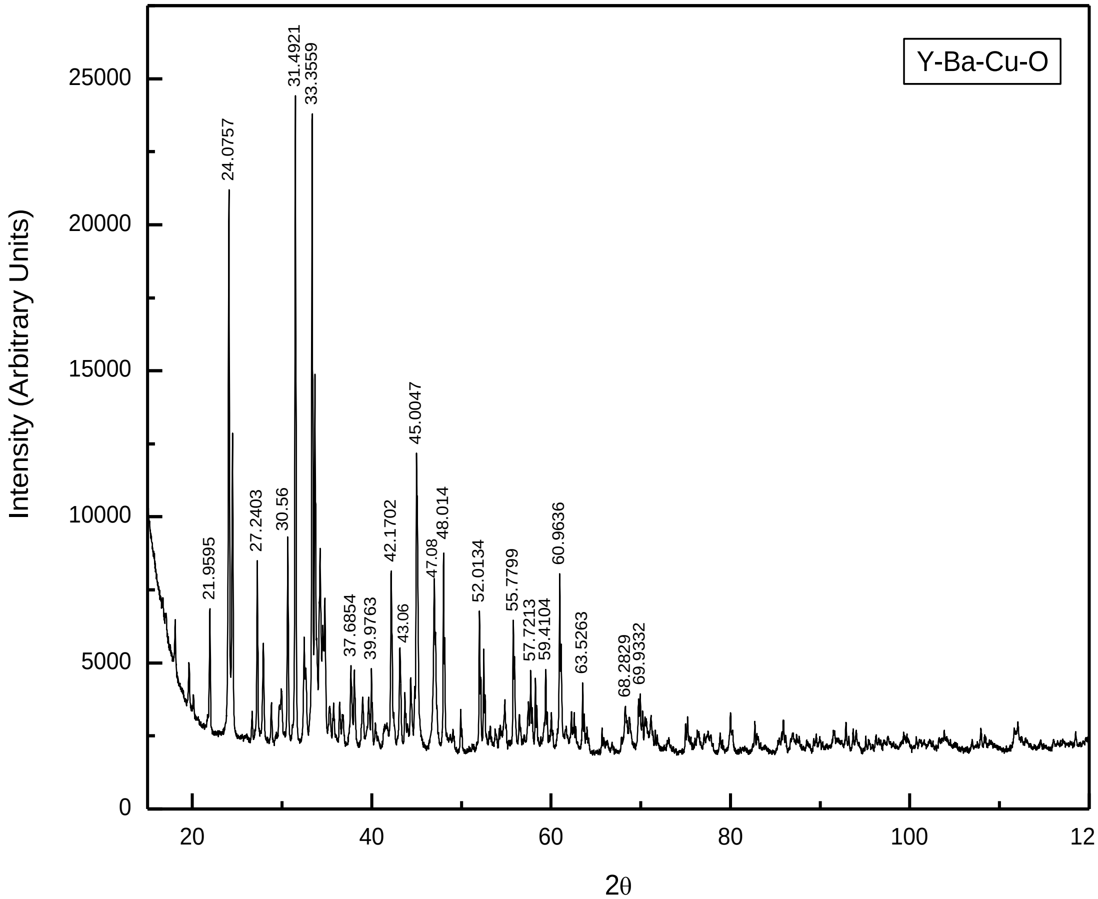

Fig 1: XRD pattern for the oxide sample.

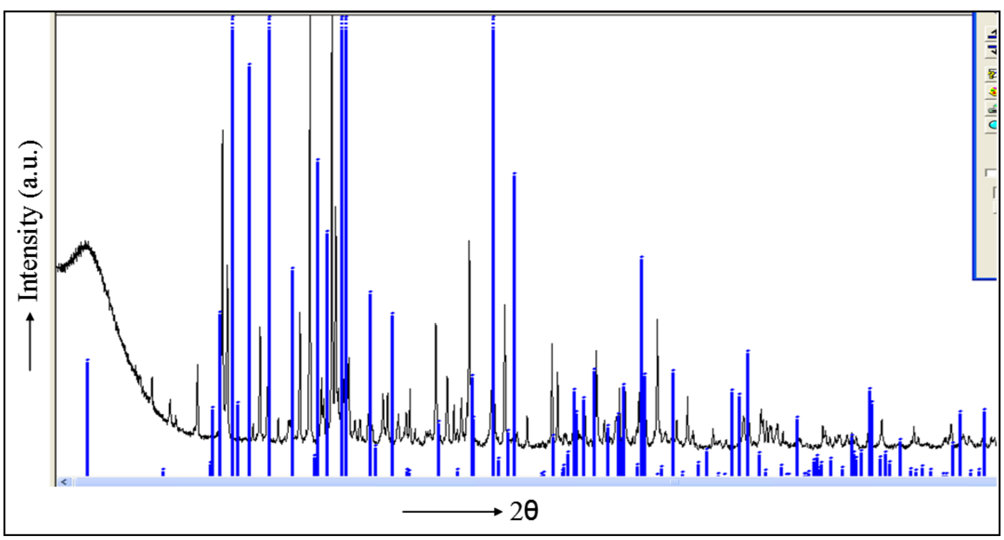

Fig 2: Sticks pattern describing matching with positions for peaks

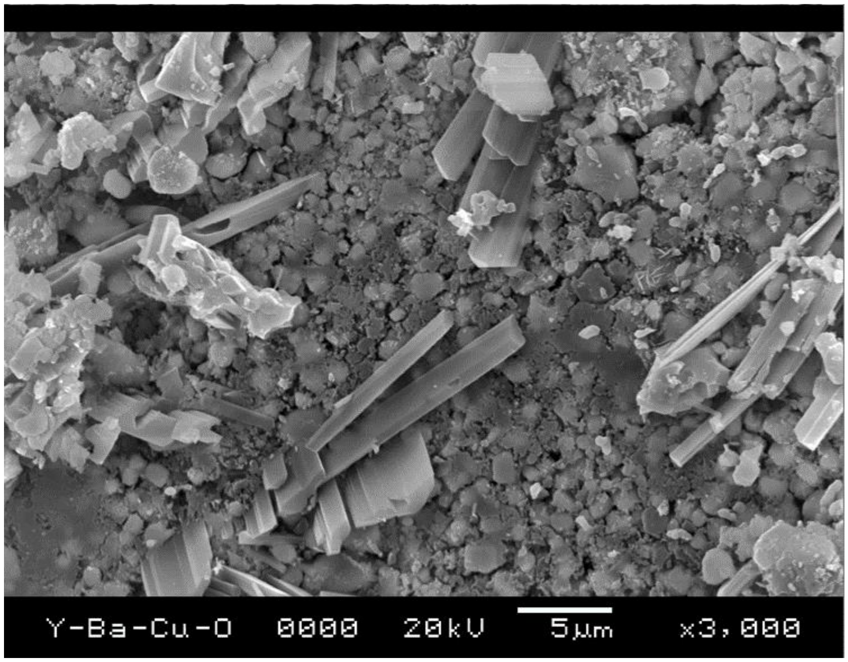

Fig 3: SEM image for the gold coated sample (X 3000 magnification).

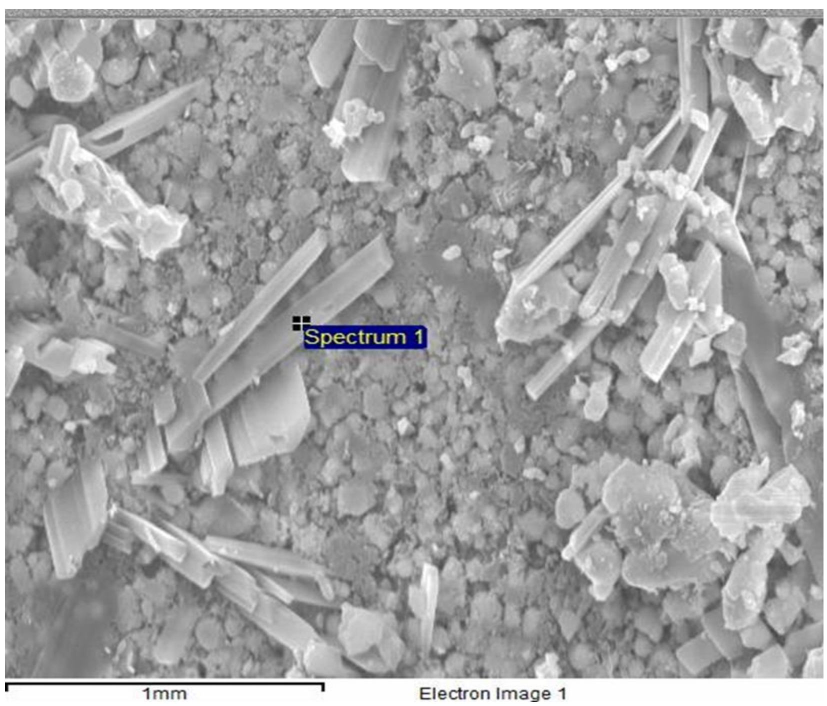

Fig 4: Rod-like phase for EDS study.

Fig 5: Spherical particle-like phase for EDS study.

Fig 6: White spherical lumps for EDS study.

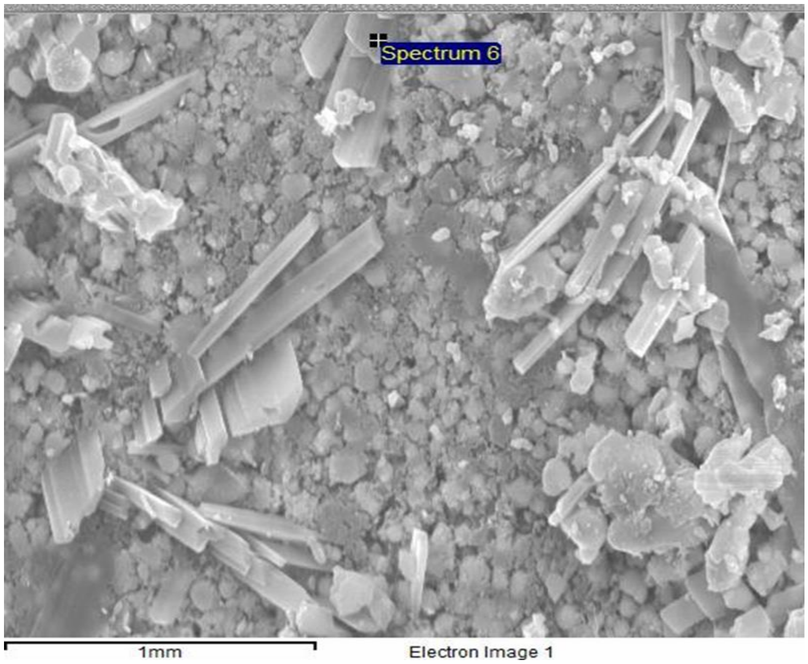

Fig 7: Broken brick block-like phase for EDS study.

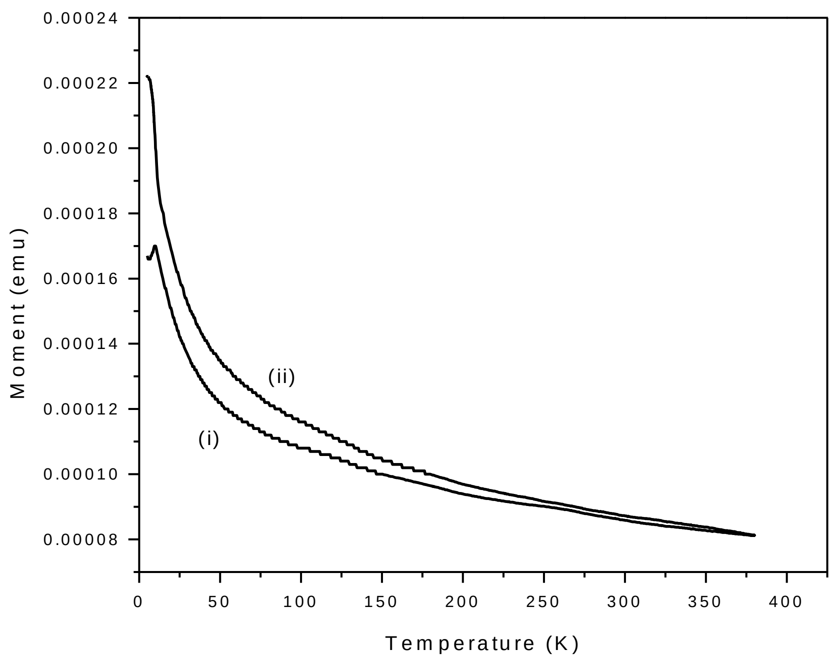

Fig 8: Moment vs. temperature plots:
(I) curve for zero field cooling and
(II) curve for field cooling.

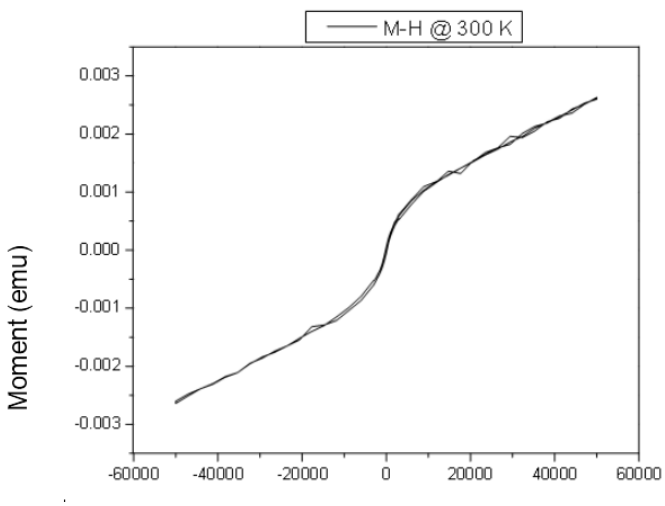

Fig 9: Moment vs. field plot at set temperature 300K.

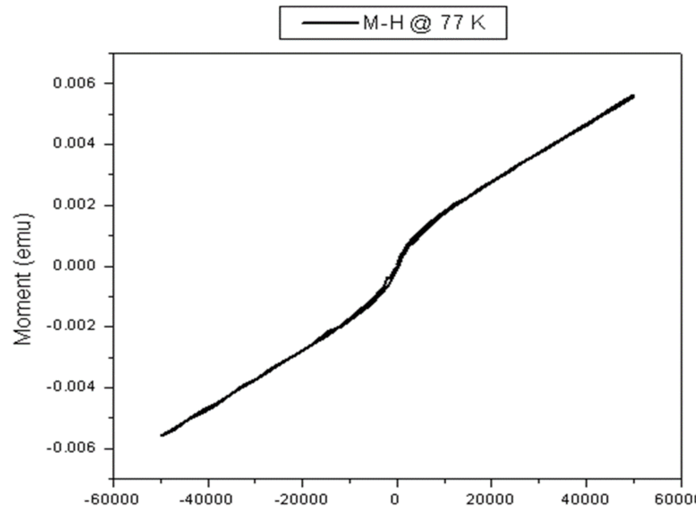

Fig 10: Moment vs. field plot at set temperature 77K.

4. Conclusion

This work is the report of characterization of $Y_1Ba_2Cu_4O_8$ prepared by polymeric precursor technique using sodium polyacrylate as the polymeric part. This work also indicates, oxide sample obtained in this process is having antiferromagnetic property. So this report is very very important. Polymeric precursor technique is expected to assist in processing of the oxide sample.

5. Acknowledgement

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6. References

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