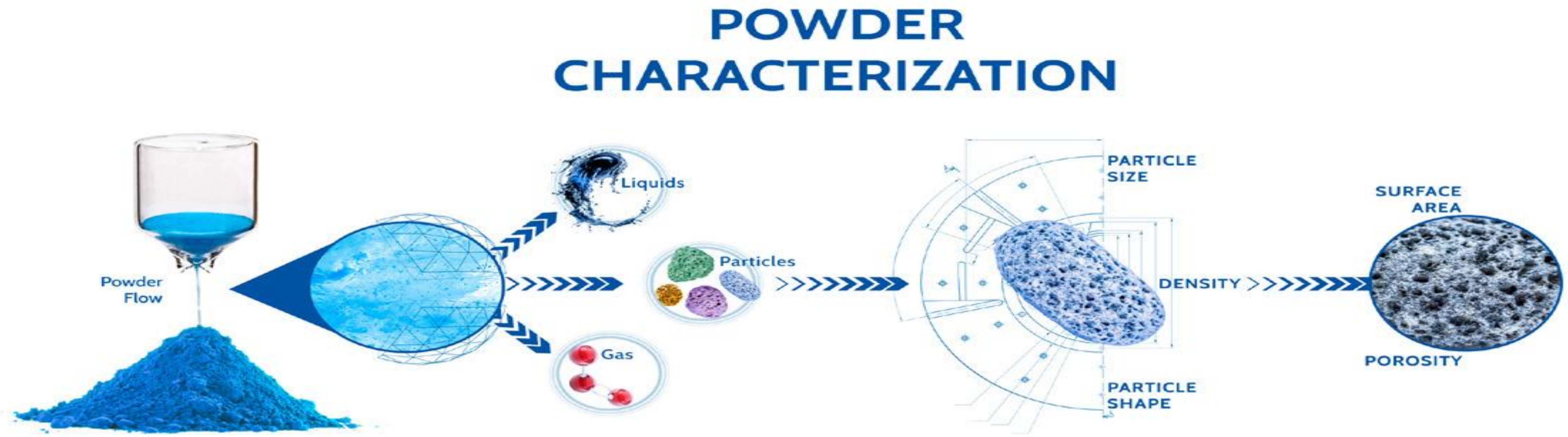
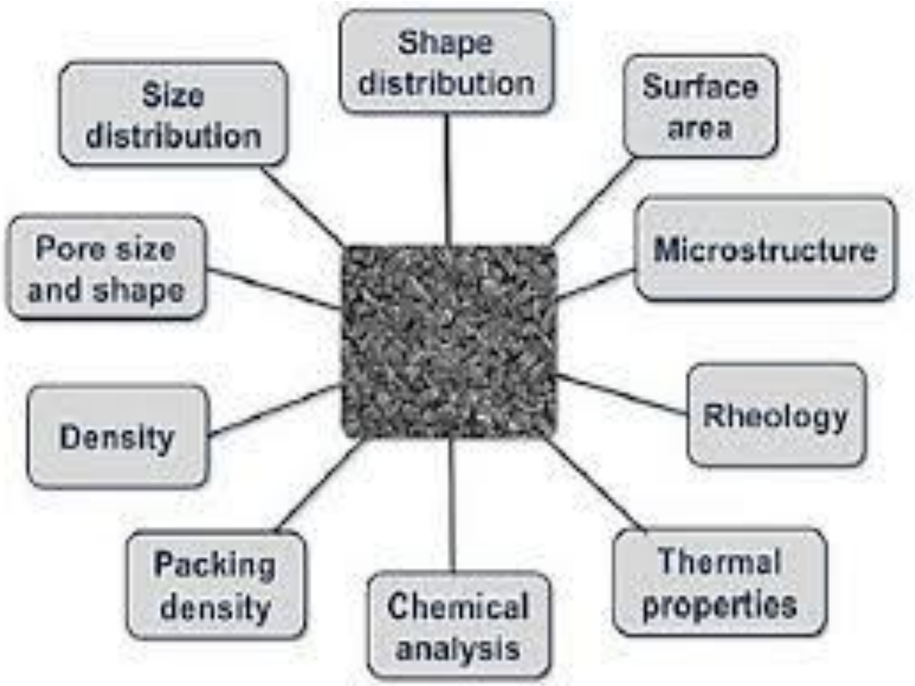
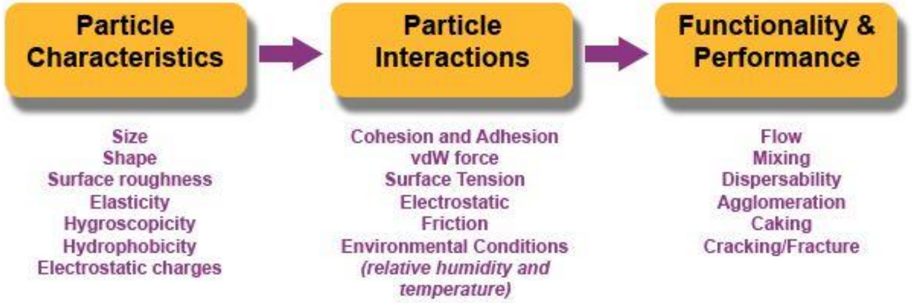


POWDER CHARACTERIZATION

1. Contents (Mechanical Engineering)

1. Introduction
2. Chemical Composition and Structure
3. Particle Size and Shape
4. Particle Surface Topography
5. Surface Area
6. Apparent and Tap Density





7. Flow Rate
8. Compressibility
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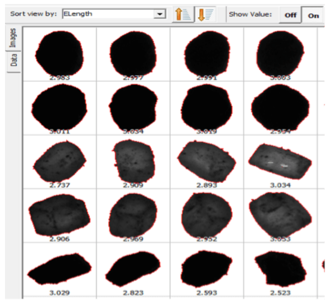
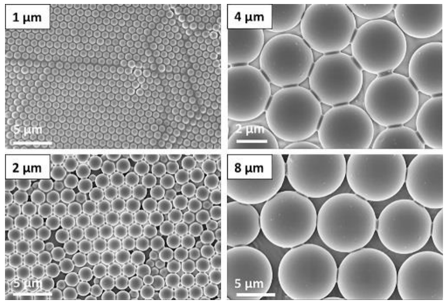
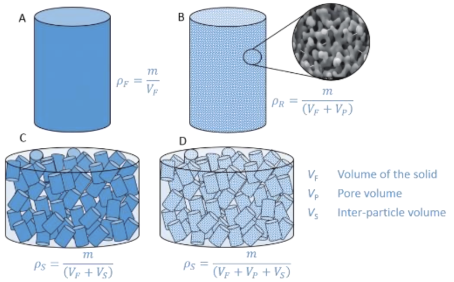
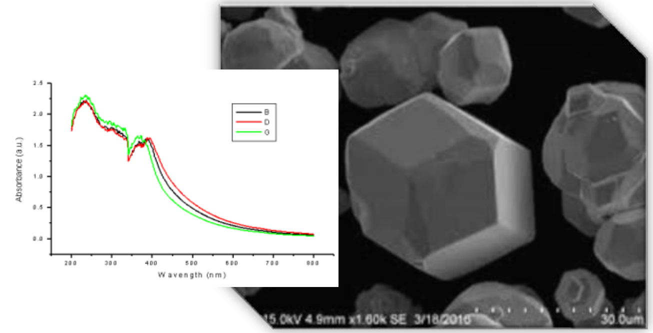




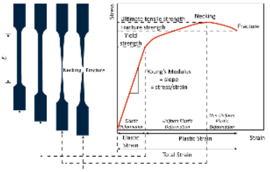


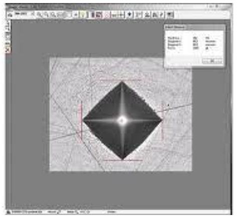
2. Contents (Material Science)



Microstructural, Chemical and Thermal Analysis

1. **XRD** (X-ray powder diffraction)
2. **XRF** (X-ray fluorescence)
3. **SEM** (Scanning Electron Microscope)
4. **EDX** (Energy-dispersive X-ray spectroscopy)
5. **DSC** (Differential Scanning Calorimeter)- **DTA** (Differential Thermal Analysis)- **TGA** (Thermo-gravimetric analysis)
6. **UV-VIS** (Ultraviolet–visible spectroscopy)





Mechanical Analysis

7. **Tension Test**
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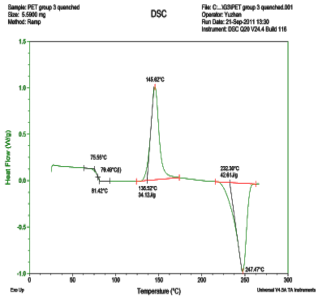
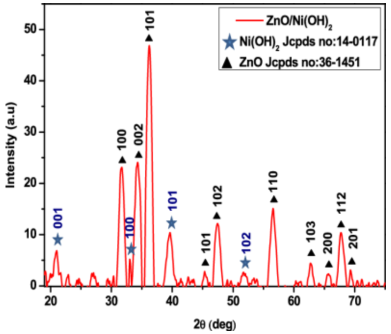
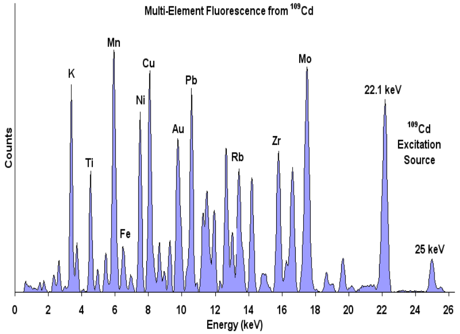
9. **Hardness (Vickers, Brinell, knoop, shore)**

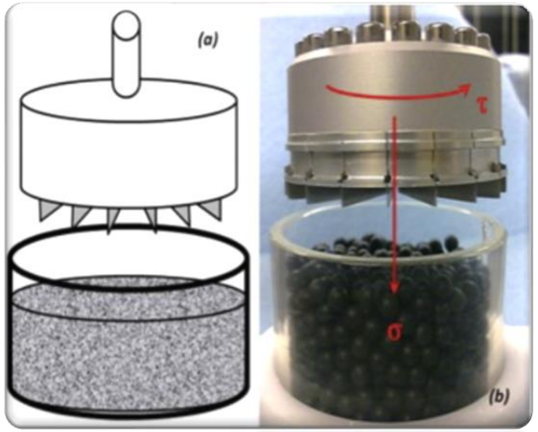


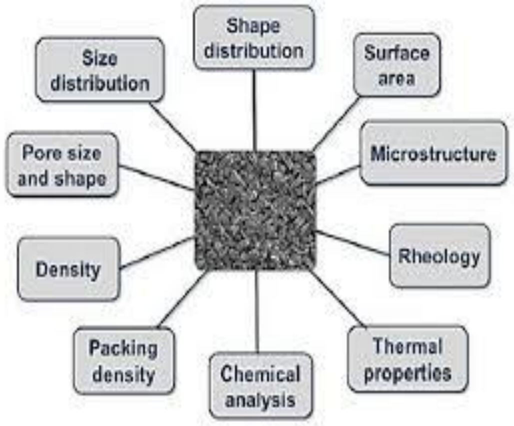


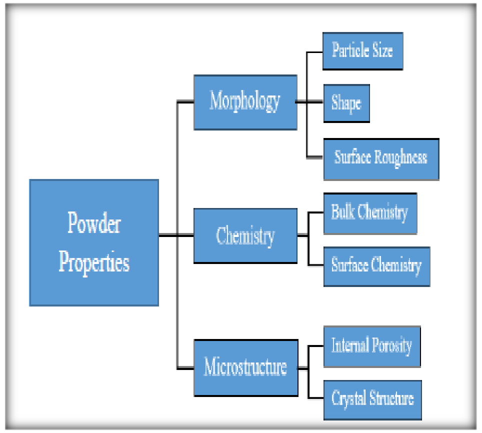


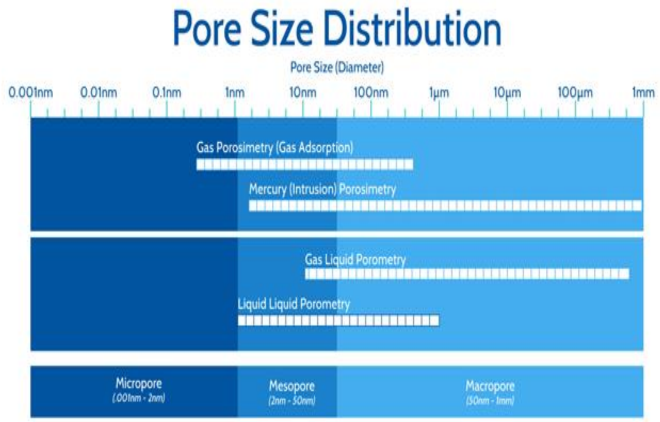
1.1.Introduction

The success of any powder metallurgical process depends to a great extent on the complete characterization and control of the metal powders. The method of powder production influences particle chemistry and structure, apart from the precise nature of particle size distribution. These properties also influence the behaviour of the powder during compaction and sintering, and the composition, structure and properties of the sintered material. In the presentation, various characteristics of the powder, some interrelated of each other, are considered. Table gives a brief summary of principles involved in the characterization of various powder properties which shall be described in subsequent sections.









1.2. Chemical Composition and Structure

Modern powder metallurgy dates only back to the early 1800

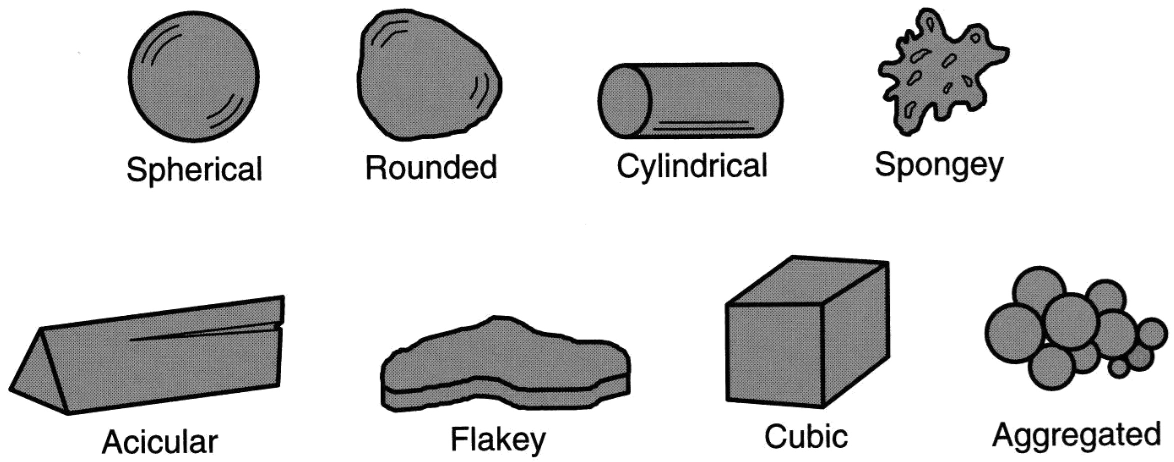
Particle size and size distribution	Sieve analysis: Permeability: Sedimentation electrical resistance; Light obscuration; Light scattering; Microscopy; Surface area.
Particle shape	SEM; Shape parameters; Morphological analysis; Fractals.
Particle shape	Stereology; Mercury Porosimetry; Gas absorption.
Particle density	Pycnometry; Mercury porosimetry.
Specific surface area	Gas absorption; Permeametry.
Surface chemistry	X-ray photoelectron spectroscopy (ESCA); Auger electron spectroscopy; Secondary ion mass spectroscopy; Ion scattering spectroscopy.
Alloy phases and phase distribution	Optical metallography; Stereology; Electron microscopy; EDAX; X-ray diffraction.
Quality of mixing [segregation]	Macroregion:Variability coefficient Microregion: Variability coefficient Homogeneity coefficient

1.2. Chemical Composition and Structure

The levels of impurity elements in metal powders can be very significant to both the processing and properties of the final product. It is necessary to know whether such elements are present in their elemental form or whether they are present in the form of a chemical compound. For example, in reduced iron powder silicon is present as impurity in the form of silica. Other ceramic and rather inert compounds may exist; these may be reported in terms of an acid insoluble figure. The effect of impurity elements on the hardness of the particles and the degree of chemical reactivity during sintering will differ widely, depending on the actual form they are in.

Metal processing technology in which parts are produced from metallic powders.

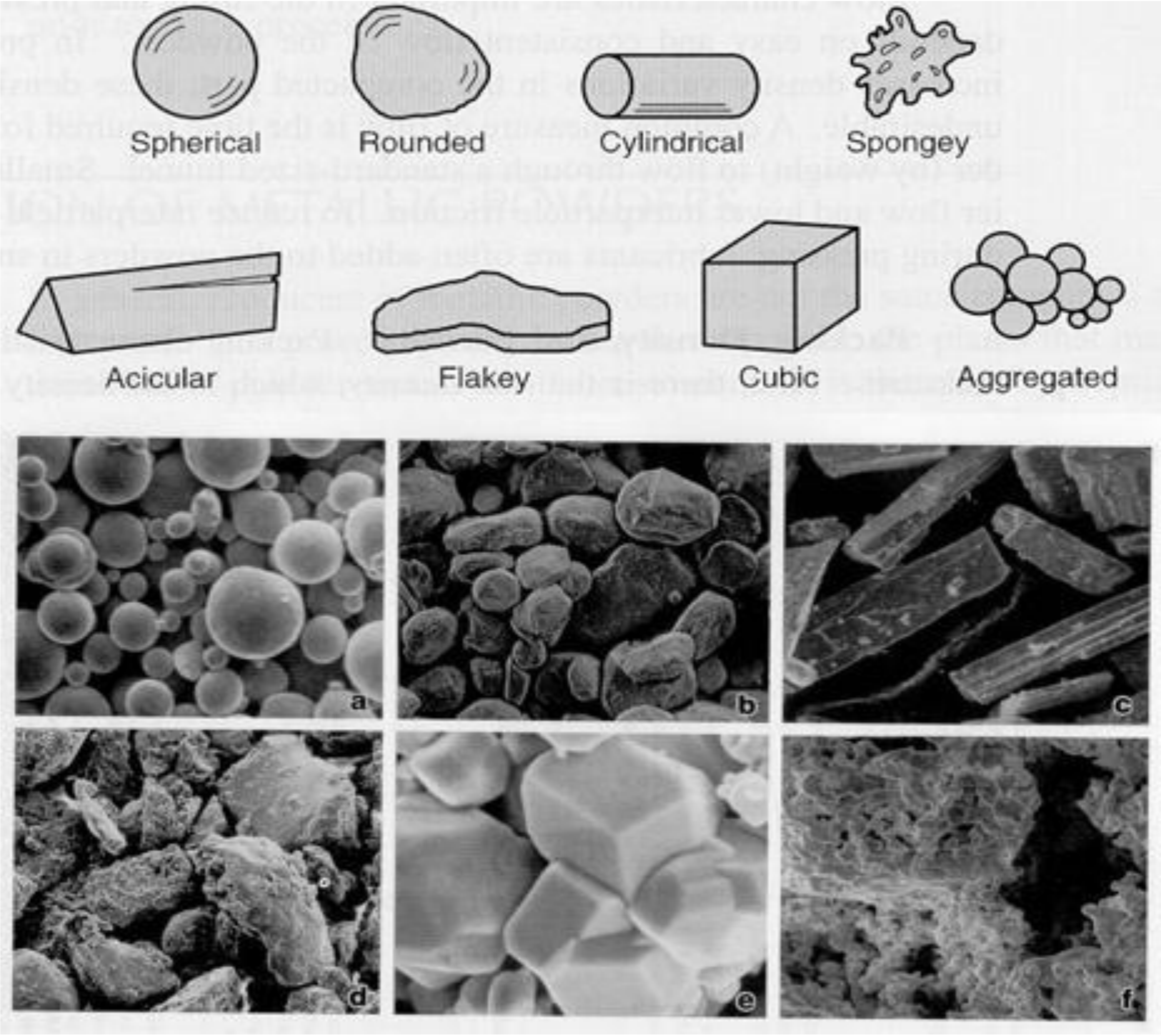
A *powder* can be defined as a finely divided particulate solid Engineering powders include metals and ceramics.



1.2. Chemical Composition and Structure

Practically any metal powder adsorbs significant quantities of gases and water vapour from the atmosphere during storage. Such adsorption can lead to the formation of surface oxides on metals which may interfere with compaction and sintering and possibly remain in the sintered material. The amount of such contamination increases with decreasing particle size and with increasing chemical activity of the surface.

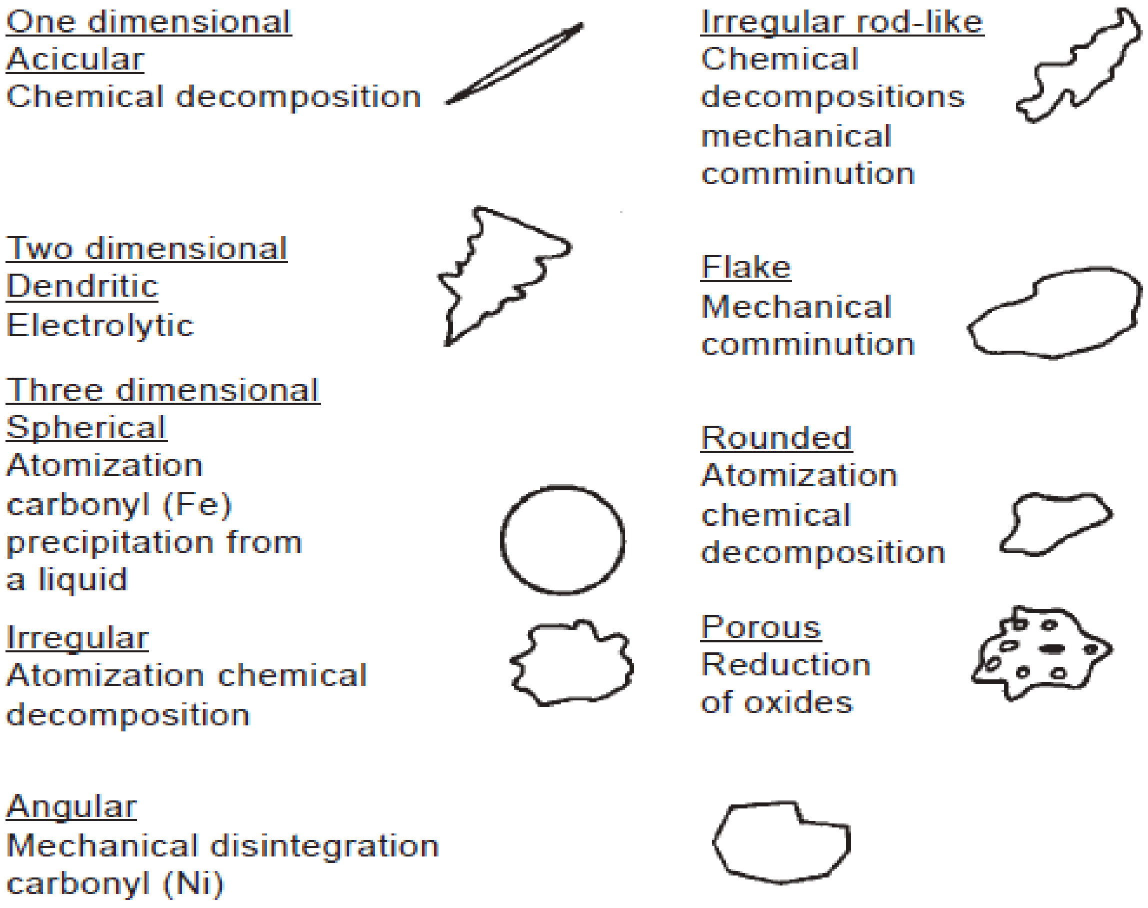
The microstructure of the crystalline powder has a significant influence on the behaviour of powder during compaction and sintering and on the properties of the final product. Fine grain size is always desirable, as it improves the mechanical properties apart from the sinterability and the uniformity of dimensional changes.



1.3. Particle Size and Shape

The shape of the powder is characterized by the dimensionality of the particle and its contour surface. An ideal system of shape characterization is given in Figure, together with the major manufacturing techniques which produce such shapes. Most powder particles are three-dimensional in nature and they may be considered as being somewhat equ-axed. Spherical particles represent the simplest and ideal example of this shape. Porous particles differ from irregular ones because of the presence of the porosity, which itself may be very irregular in both size and shape. A large amount of porosity makes any shape characterization very difficult.

System of particles shape characterization.



1.3. Particle Size and Shape

There are a number of particle size measurement techniques available in powder metallurgy, each having their own limitations. Table classifies some of the common methods of particle size determination and their limits of applicability.

Class	Method	Approximate useful size range (microns)
Sieving	Sieving using mechanical agitation or ultrasonic induced agitation and screens	44–800
Microscopy	Micromesh screens Visible light Electron microscopy	5–50 0.2–100 0.001–5
Sedimentation	Gravitational Centrifugal	1–250 0.05–60
Turbidimetry	Turbidimetry (light intensity attenuation measurements)	0.05–500
Elutriation	Elutriation	5–50
Electrolytic resistivity	Coulter counter	0.5–800
Permeability	Fisher sub-sieve sizer	0.2–50
Surface area	Adsorption from gas phase Adsorption from liquid phase	0.01–20 0.01–50

1.3. Particle Size and Shape

Out of all the methods, sieving is technologically most satisfactory for reporting and plotting particle size distribution, in which the successive sizes form a geometrical series. The reference point for their scale has become 75 micrometer which is the opening of the 200-mesh woven wire screen standardized by the National Bureau of Standards. Table gives some of the most pertinent data for both the Tyler standard and United States sieve series. Micromesh sieve are also available with openings down to at least five micrometers, but are rather difficult to use and maintain, are very fragile and have low load capacities. These sieves are produced by electrodeposition of nickel or copper on to photosensitized machine ruled lines.

Another advantage of screening is that one may synthesize a desired distribution, according to the type of blend required by the manufacturer. Conventional sieving requires a sample of 50 g for accurate analysis and this becomes difficult in analysis of expensive metal powders.

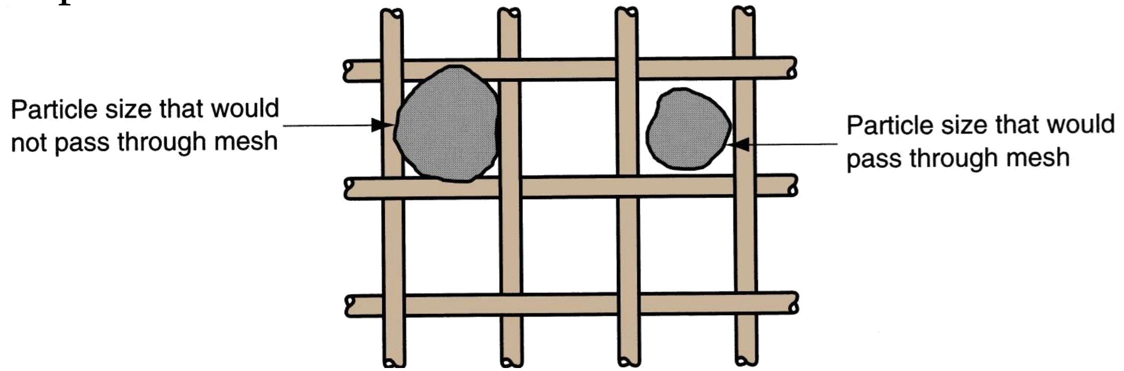


Figure 1. Screen mesh for sorting particle sizes.

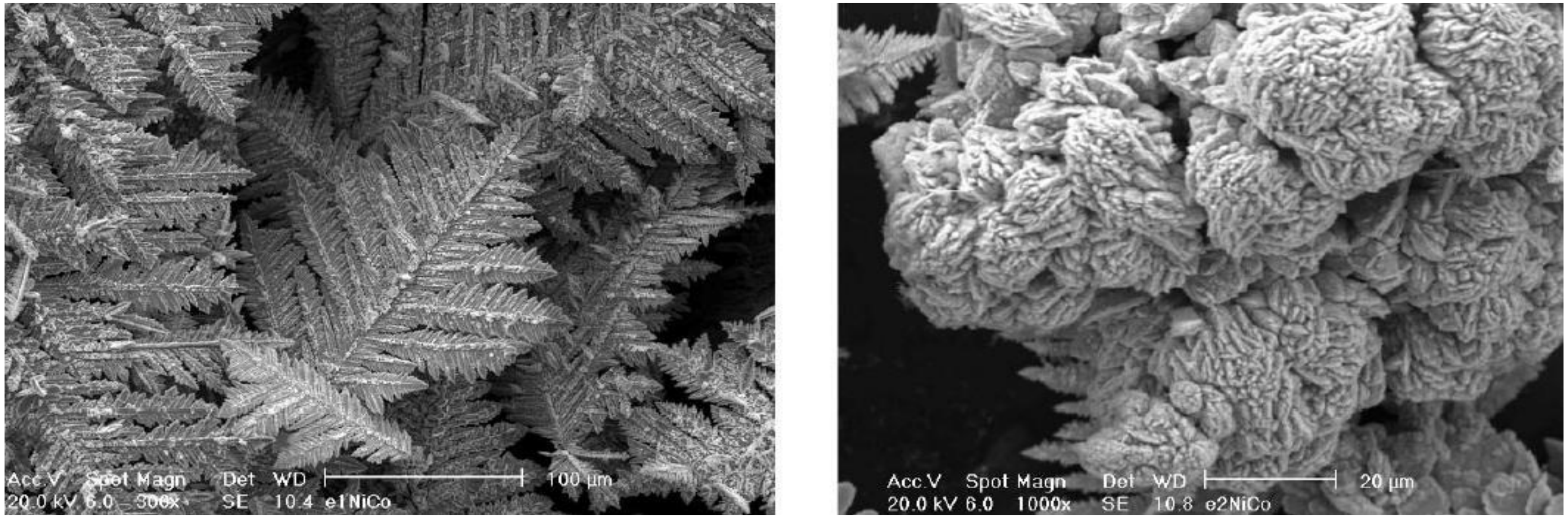
1.3. Particle Size and Shape

Mesh designation number	Sieve opening (μm)		
	New US series	Old US series	Tyler series
20	850	841	833
35	–	–	417
40	425	420	–
60	250	250	295
80	180	177	175
100	150	149	147
140	106	105	–
150	–	–	104
200	75	72	74
230	63	63	–
250	–	–	63
325	45	44	44

Figure 2. Information on sieves used in powder metallurgy

1.4. Particle Surface Topography

Figure 3. Ni/Co powder



The nature of the surface of the individual particles is also an important powder characteristic. A spherical particle may appear smooth, but on a closer examination at high magnifications the surface may actually consist of many protuberances. Reduced metal powder has a highly roughened surface.

Atomized metal powders, on the other hand, have finer degree of surface roughness, which are of rounded type rather than sharp and irregular. Scanning electron microscope is a powerful tool for examining surface topography.

1.5. Surface Area

The actual amount of surface area per unit mass of powder is of great significance. Any reaction between the particles or between the powder and its environment starts at these surfaces. This affects sinterability. For a very irregular shaped particle with a high degree of surface roughness, the specific surface area can be very high. The surface area of a given powder is measured by the BET method, in which an adsorption of a species in solution may be used to obtain a value of specific surface (*SW*) if the surface is completely covered by a monomolecular layer of the solute. From a knowledge of the area occupied by one molecule, the total area of the powder sample and, finally, *SW* can be obtained.

The amount of gas adsorbed in a monomolecular layer in m^2 is calculated from an adsorption isotherm, i.e. a series of measurements of the volume V of gas adsorbed as a function of pressure p.

The BET method of determining the specific surface is widely used for catalysts. Its use for metal powder is primarily for very fine powders, particularly those of the refractory metals and for characterizing the total surface area of porous powders.

1.6. Apparent and Tap Density

The apparent density of a powder refers to the mass of unit volume of loose powder usually expressed in g/cm^3. It is one of the most critical characteristics of a powder, because of following reasons:

(*a*) It determines the size of the compaction tooling and the magnitude of press motions necessary to compact and densify the loose powder;

(*b*) It determines the selection of equipment used to transport and treat the initial powder;

(*c*) It influences the behaviour of the powder during sintering Other characteristics which have direct bearing on apparent density are the density of the solid material, particle size and shape, surface area, topography and its distribution. Apparent density is determined by the Hall flowmeter, where a container of known volume (25 ml) is completely filled by flowing metal powder through a Hall funnel (Figure 4)

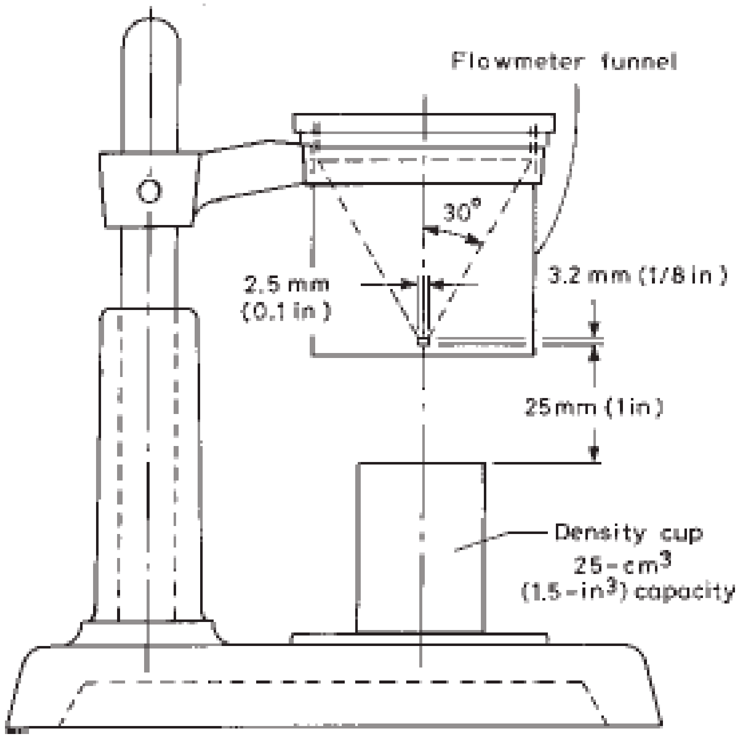


Figure 4. Hall flowmeter.

1.7. Flow Rate

Rapid rates of P/M parts production require a relatively rapid flow of powder from storage containers to dies. The standard method for its determination is by the Hall flowmeter, where the time necessary for 50 g of powder to flow through a prescribed small orifice is measured. The test offers only a means of comparison and evaluation because in the majority of operation conditions the powder does not have to flow through a small orifice. Flow times are, therefore, proportional to the reciprocal of the flow rates. Very fine powders do not flow through a small orifice. This is a result of the drastic increase in the specific surface area as the size becomes very small.

Material	Apparent density (g/cc)	Tap density (g/cc)	Percent increase
Copper (a)			
spherical	4.5	5.3	18
irregular	2.3	3.14	35
flake	0.4	0.7	75
Iron (–100+200 mesh)			
electrolytic	3.31	3.75	13
atomized	2.66	3.26	23
sponge	2.29	2.73	19
Aluminium (–200 mesh)			
atomized	0.98	1.46	49

(a) all copper powders with same size distribution; from H.H. Hausner, in: Handbook of Metal Powders, A.R.Poster, editor, Reinhold, N.Y., 1966.

Figure 5. Apparent and tap densities of various powders(2)

1.8. Compressibility

Compressibility is a measure to which a powder will compress or densify upon application of external pressure. Compressibility is reported as the density in g/cm^3, rounded to the nearest 0.01 g/cm^3, at a specified compaction pressure, or as the pressure needed to reach a specified density. Typically, a cylinder or rectangular test piece is made by pressing powder in a die, with pressure applied simultaneously from top and bottom.

Compressibility of the powder is influenced by factors like: inherent hardness of the concerned metal or alloy, particle shape, internal porosity, particle size distribution, presence of non-metallics, addition of alloying elements
or solid lubricants Compressibility, alternatively, is defined in terms of the densification parameter, which is equal to:

$$\text{Densification parameter} = \frac{\text{Green density} - \text{Apparent density}}{\text{Theoretical density} - \text{Apparent density}}$$

1.8. Compressibility

Compressibility, in general, increases with increasing apparent density. A rather large amount of densification occurs at relatively low compaction pressure. Another term, which is very important for tooling design, is the compression ratio. It is the ratio of the volume of loose powder to the volume of the compact made from it. A low compression ratio is desirable because of following reasons:

– Size of the die cavity and tooling can be reduced

– Breakage and wear of tooling is reduced

– Press motion can be reduced

– A faster die fill and thus a higher production rate can be achieved.

Powder	Apparent density (g/cm³)	Compaction pressure		Green density (g/cm³)	Green strength	
		(N/mm²)	(tsi)		(psi)	(N/mm²)
Sponge [a]	2.4	415	30	6.2	14.41	2100
		550	40	6.6	22.05	3200
		690	50	6.8	28.25	4100
Atomized sponge [b]	2.5	414	30	6.55	13.09	1900
		550	40	6.8	18.80	2700
		690	50	7.0		
Reduced [a]	2.5	415	30	6.5	15.85	2300
		550	40	6.7	20.67	3000
		690	50	6.9	24.11	3500
Sponge [a]	2.6	415	30	6.6	18.60	2700
		550	40	6.8	24.80	3600
		690	50	7.0	26.87	3900
Electro [c]	2.6	415	30	6.3	31.69	4600
		550	40	6.7	42.72	6200
		690	50	6.95	53.74	7800

[a] powders contained 1% zinc sterate blended in
[b] powders contained 0.75% zinc stearate blended in
[c] unlike the other powders, this one was isostatically pressed (from C.E.Buren and H.H.Hirsch, in: Powder Metallurgy, Interscience, New York, 403–440)

Figure 6. Green density and green strength for various types of iron powders

1.9. Green Strength

Green strength is the mechanical strength of a green – i.e. un-sintered powder compact. This characteristic is very important, as it determines the ability of a green compact to maintain its size and shape during handling prior to sintering.

Green strength is promoted by:

– increasing particle surface roughness, since more sites are available for mechanical interlocking;

– increasing the powder surface area. This is achieved by increasing the irregularity and reducing the particle size;

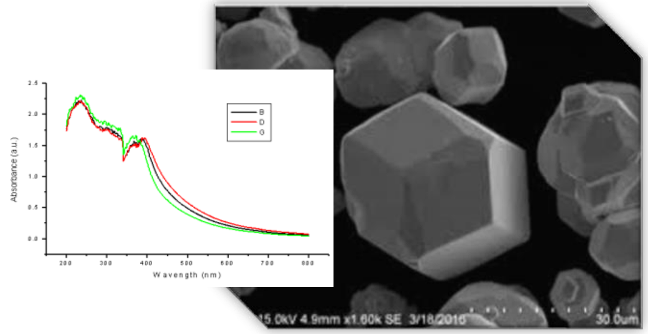
– decreasing the powder apparent density. This is a consequence of first two factors;

– decreasing particle surface oxidation and contamination;

– increasing green density (or compaction pressure);

– decreasing the amount of certain interfering additives. For example, the addition of small alloying elements, such as soft graphite to iron and lubricant, prevents mechanical interlocking.

2. Contents (Material Science)



Microstructural, Chemical and Thermal Analysis

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2. **XRF** (X-ray fluorescence)
3. **SEM** (Scanning Electron Microscope)
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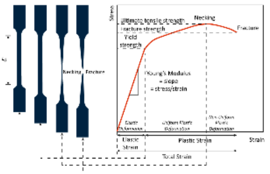
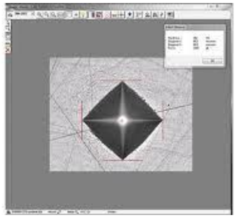
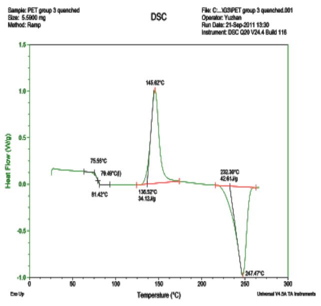
6. **UV-VIS** (Ultraviolet–visible spectroscopy)

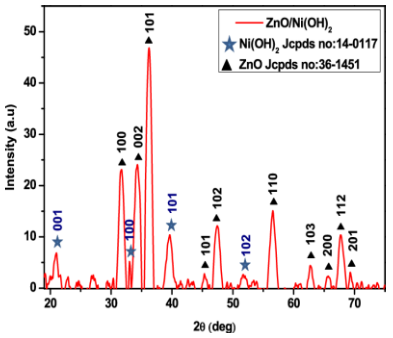


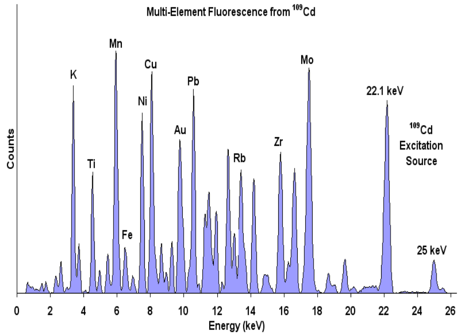


Mechanical Analysis

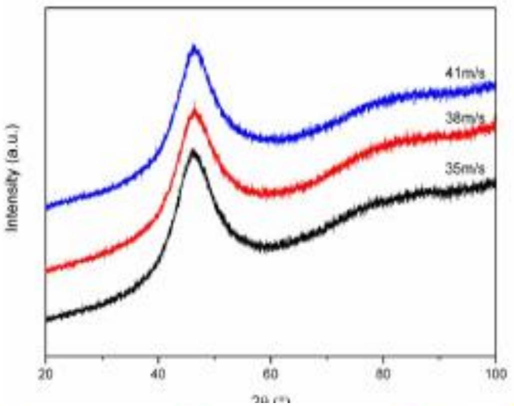
7. **Tension Test**
8. **Compression Test**
9. **Hardness (Vickers, Brinell, knoop,shore)**

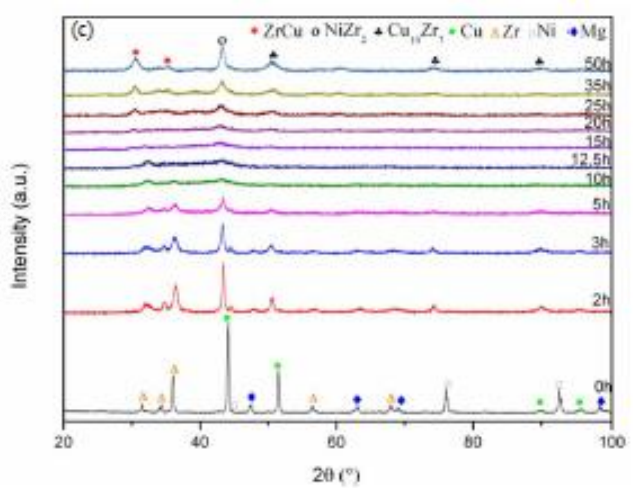


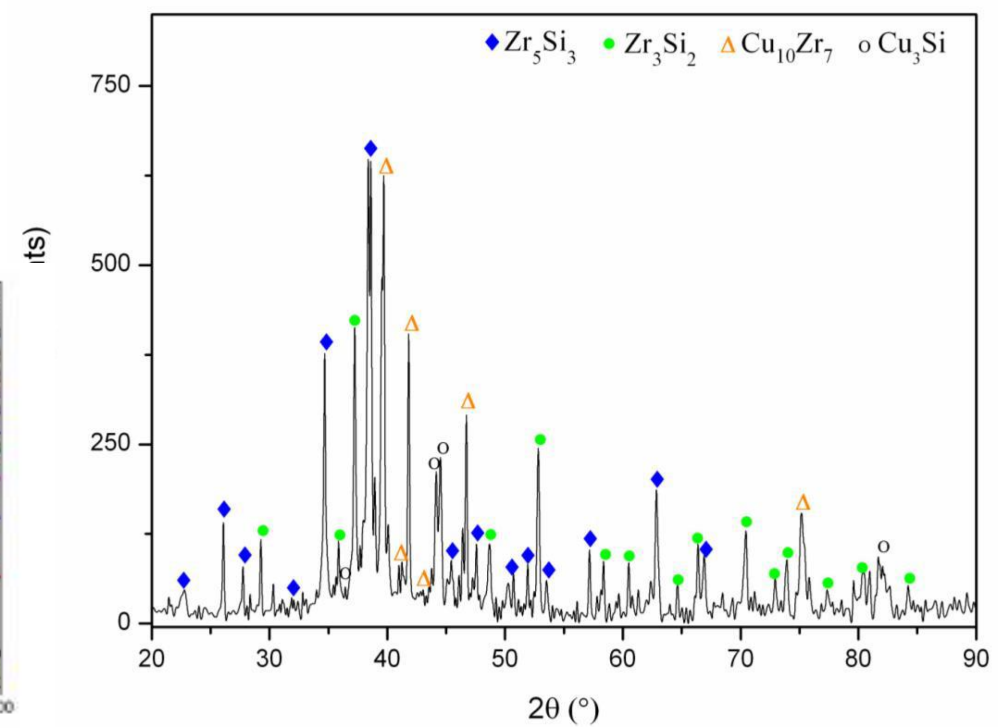


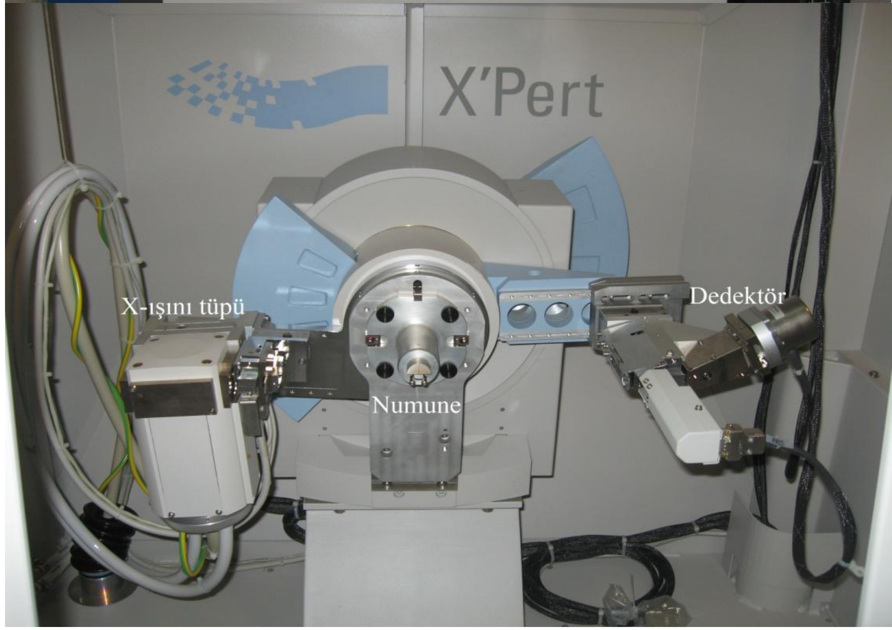


2.1. XRD (X-ray powder diffraction)

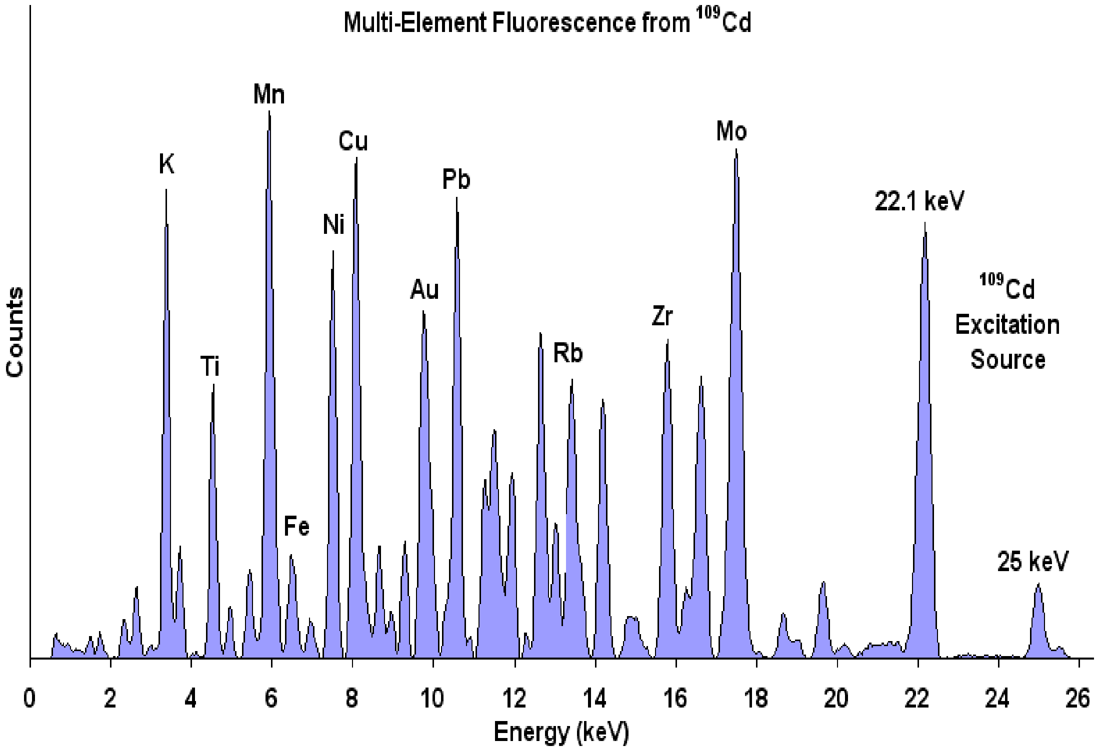


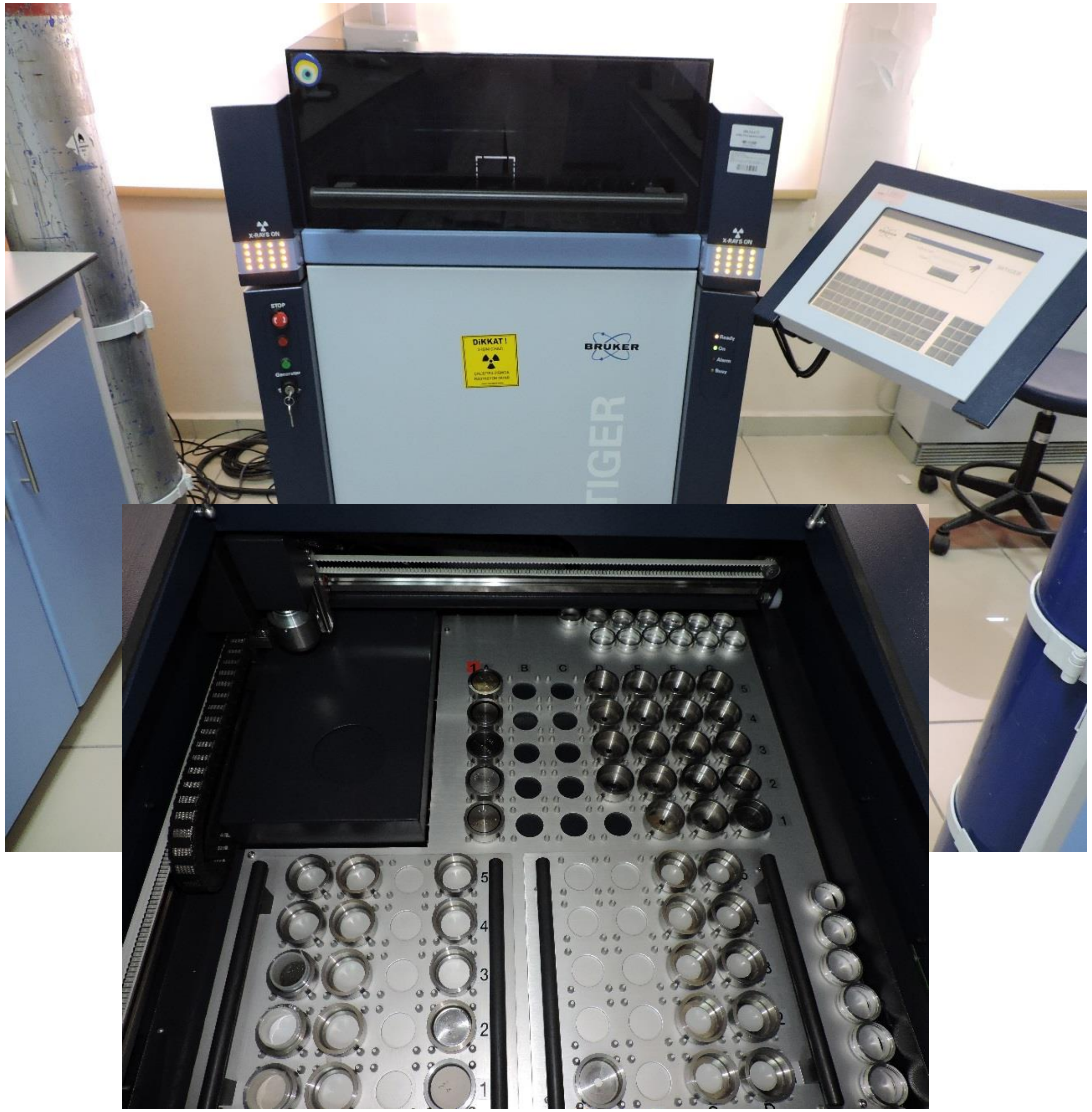




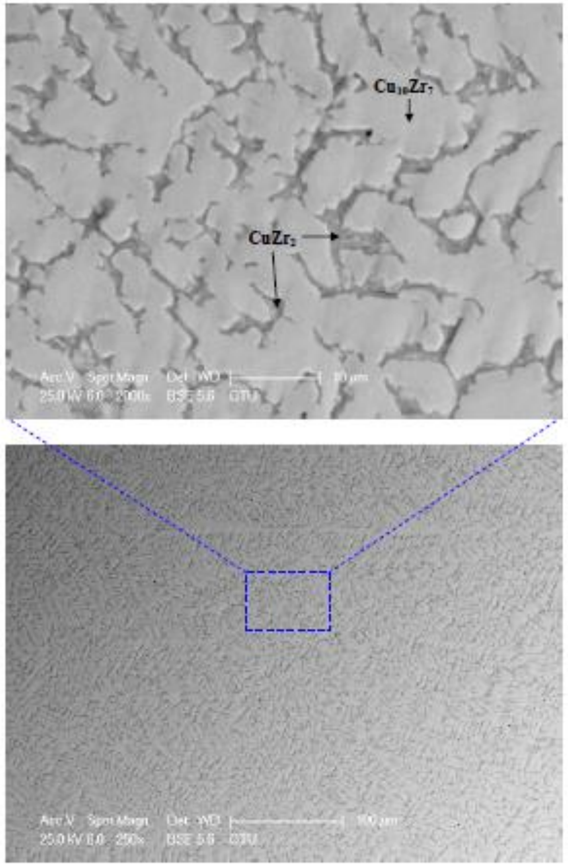


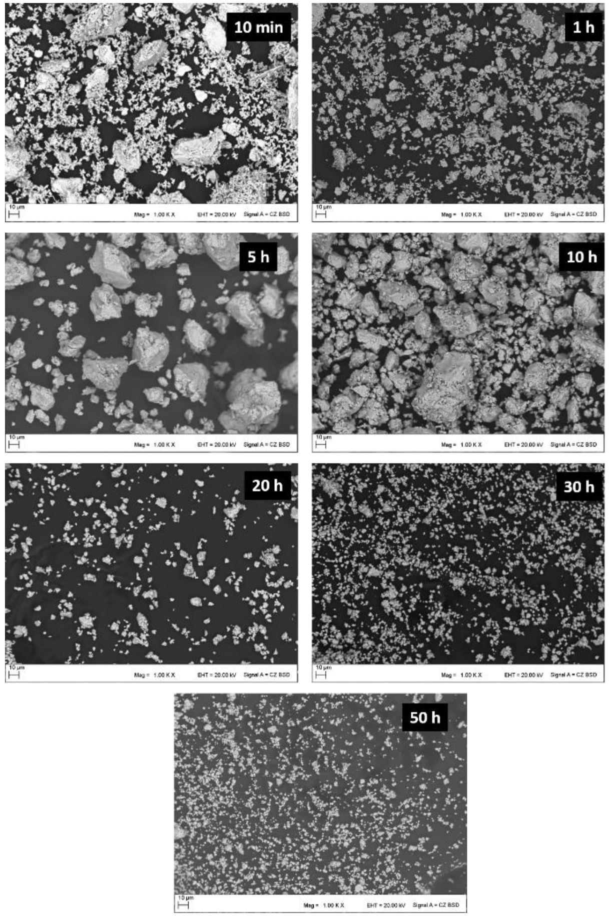
2.2. XRF X-ray fluorescence)



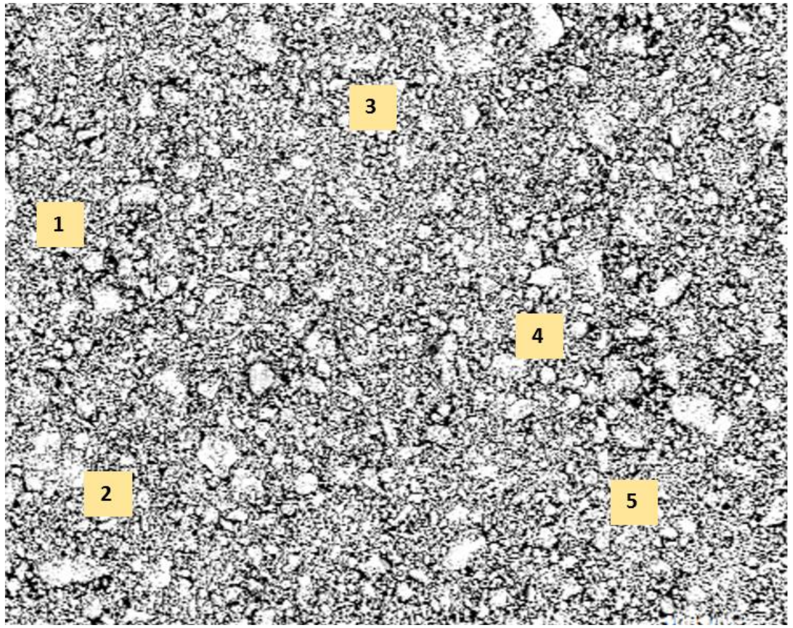


2.3. SEM(Scanning Electron Microscope)

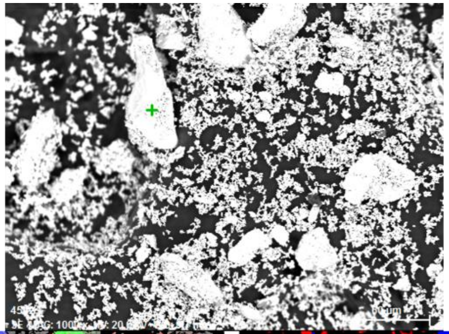


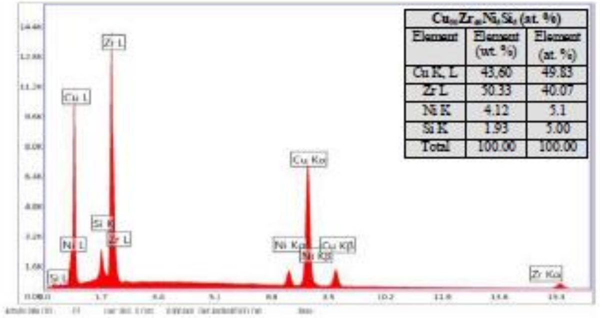


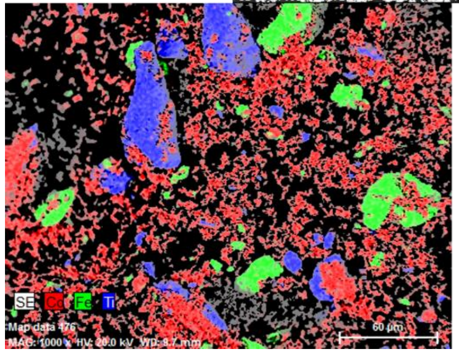
2.4. EDX

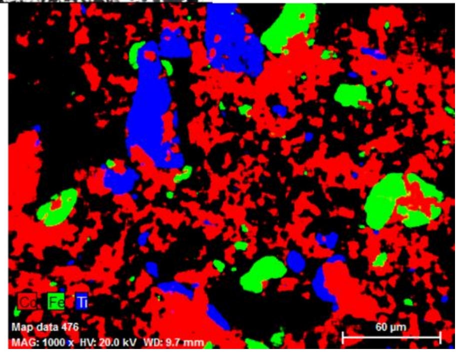


Pellet	Elements of Compounds - Atom. C [at.%]			
Spectrum	Co	Fe	Ti	B
1	35.38	40.35	10.15	14.12
2	29.71	34.05	8.51	27.73
3	34.71	39.84	10.08	15.37
4	37.02	42.54	10.87	9.57
5	31.31	36.72	8.99	22.98

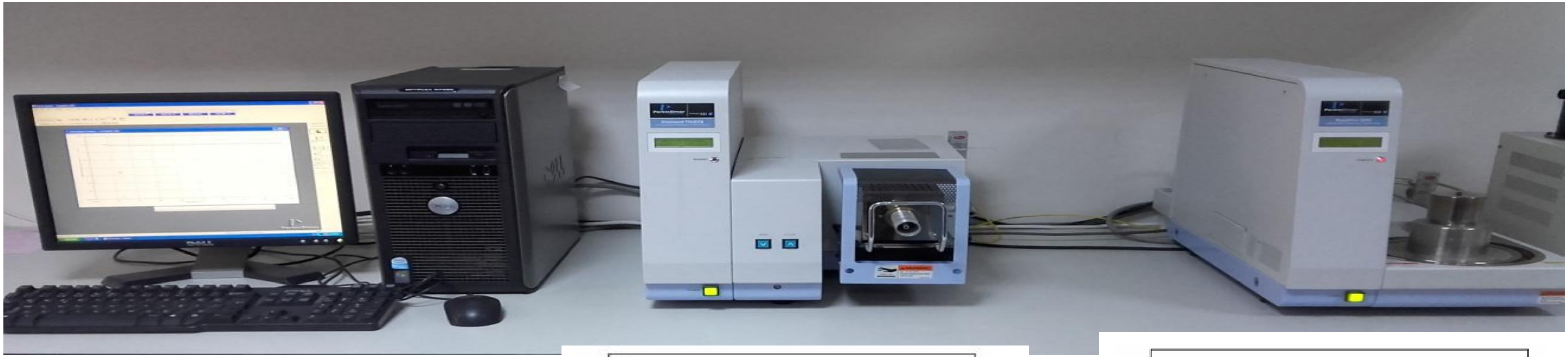


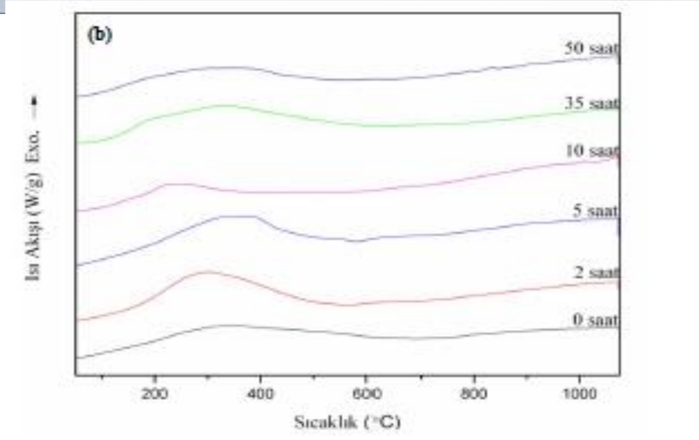


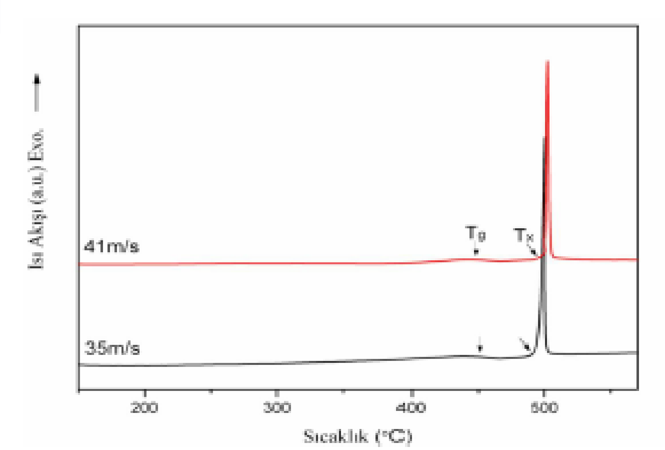




2.5. DSC-DTA-TGA







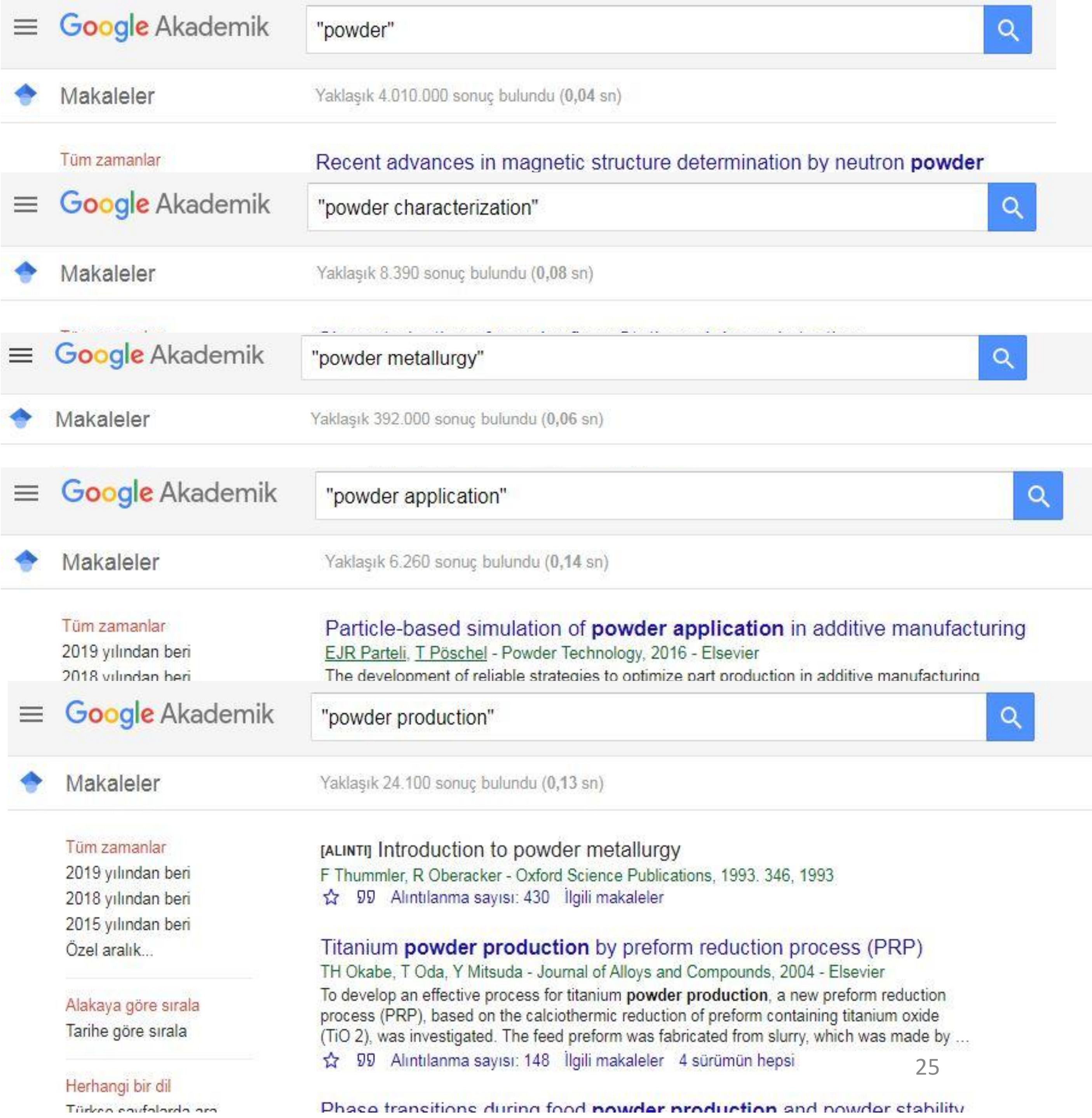
Powder = 4.010.000

Powder metallurgy = 392.000

Powder production = 24.100

Powder characterization = 8390

Powder application = 6260



Reference

1. R.M. German, Powder Metallurgy Science, 2nd edition, MPIF, Princeton, 1994.
2. J.S. Hirschhorn, Introduction to Powder Metallurgy, American Powder Metallurgy Institute, New York, 1969.
3. Fundamentals of powder metallurgy by W. D. Jones
4. Powder Metallurgy: Principles and Applications by F. V. Lenel
5. Fundamentals of P/M by I. H. Khan

Thanks For Listening