2- POWDER SYNTHESIS METHODS

2-1 INTRODUCTION:

A variety of methods exist for the synthesis of ceramic powders. In this book, we divide them into two categories: *mechanical methods* and *chemical methods*.

Mechanical methods are generally used to prepare powders of traditional ceramics from naturally occurring raw materials. Powder preparation by mechanical methods is a fairly mature area of ceramic processing in which the scope for new developments is rather small. However, in recent years, the preparation of fine powders of some advanced ceramics by mechanical methods involving milling at high speeds has received a fair amount of interest.

Chemical methods are generally used to prepare powders of advanced ceramics from synthetic materials or from naturally occurring raw materials that have undergone a considerable degree of chemical refinement. Some of the methods categorized as chemical involve a mechanical milling step as part of the process. The milling step is usually necessary for the breakdown of agglomerates and for the production of the desired physical characteristics of the powder such as average particle size and particle size distribution. Powder preparation by chemical methods is an area of ceramic processing that has seen several new developments in the past 25 years and further new developments are expected in the future. Table 2.1 provides a summary of the common powder preparation methods for ceramics

2-2 POWDER SYNTHESIS BY CHEMICALMETHODS

A wide range of chemical methods exist for the synthesis of ceramic powders and several reviews of the subject are available in the ceramic literature. For convenience, we will consider the methods in three fairly broad categories :(1) solid-state reactions, (2) synthesis from liquid solutions, and (3) vapor-phase reactions.

TABLE 2.1 Common Powder Preparation Methods for Ceramics

Powder preparation method	Advantages	Disadvantages
Mechanical		
Comminution	Inexpensive, wide applicability	Limited purity, limited homogeneity, large particle size
Mechanochemical synthesis	Fine particle size, good for nonoxides, low temperature route	Limited purity, limited homogeneity
Chemical		
Solid-state reaction		
Decomposition, reaction between solids	Simple apparatus, inexpensive	Agglomerated powder, limited homogeneity for multicomponent powders
Liquid solutions		
Precipitation or coprecipitation; solvent vaporization (spray drying, spray pyrolysis, freeze drying); gel routes (sol–gel, Pechini, citrate gel, glycine nitrate)	High purity, small particle size, composition control, chemical homogeneity	Expensive, poor for nonoxides, powder agglomeration commonly a problem
Nonaqueous liquid reaction	High purity, small particle size	Limited to nonoxides
Vapor-phase reaction		
Gas–solid reaction	Commonly inexpensive for large particle size	Commonly low purity, expensive for fine powders
Gas–liquid reaction	High purity, small particle size	Expensive, limited applicability
Reaction between gases	High purity, small particle size, inexpensive for oxides	Expensive for nonoxides, agglomeration commonly a problem

2-2.1 Solid-State Reactions

Chemical decomposition reactions, in which a solid reactant is heated to produce a new solid plus a gas, are commonly used for the production of powders of simple oxides from carbonates, hydroxides, nitrates, sulfates, acetates, oxalates, alkoxides, and other metal salts. An example is the decomposition of calcium carbonate (calcite) to produce calcium oxide and carbon dioxide gas:

$$CaCO_3(s) \rightarrow CaO(s) + CO_2(g) \qquad (2\text{-}1)$$

Chemical reactions between solid starting materials, usually in the form of mixed powders, are common for the production of powders of complex oxides such as titanates, ferrites, and silicates. The reactants normally consist of simple oxides, carbonates, nitrates, sulfates, oxalates, or acetates. An example is the reaction between zinc oxide and alumina to produce zinc aluminate:

$$ZnO(s) + Al_2O_3(s) \rightarrow ZnAl_2O_4(s) \quad (2\text{-}2)$$

These methods, involving decomposition of solids or chemical reaction between solids are referred to in the ceramic literature as *calcination*.