In the present investigations, the Mossbauer effect studies of the thermal decomposition in hydrogen of ferrous oxalate and of homogeneously mixed ferrous-nickel oxalate of different Fe:Ni ratios are carried out. By carrying out thermal decomposition in hydrogen, simultaneous decomposition and reduction of the sample takes place. The products are formed at lower temperatures of decomposition and in shorter durations than in the conventional methods. It is possible to get the metal and alloy powders directly without an intermediate stage of an oxide phase. Whenever conditions are not favourable and product gasses accumulate, oxide and carbide phases appear.

Superparamagnetic fine particles are initially formed during the decomposition and reduction. Due to the fineness of the particles they are very pyrophoric. To avoid oxidation during exposure to atmosphere, careful steps are taken to pretreat the samples. The ESCA studies confirm that less than six layers of oxygen are formed on the surface of the product sample rendering them non-pyrophoric. The stoichiometry of the sample in alloy formation does not exactly follow that of mixed oxalates taken as indicated for example by Invar formation.
during the decomposition of mixed metal oxalate with Fe to Ni ratio of 1:1. The Mossbauer studies show that equal amounts of iron and nickel are most favourable for the formation of Invar, in the mixed metal oxalates.

The Mossbauer effect is used as a fingerprint technique to follow the mechanism of thermal decomposition of ferrous oxalate and mixed metal oxalates in hydrogen. Initially, Fe$_3$O$_4$ particles are formed when ferrous oxalates is decomposed in hydrogen. The presence of superparamagnetic Fe$_3$O$_4$ particles is confirmed by low temperature Mossbauer studies. An estimate of the size of particles gives a value of 162Å for the particles obtained when ferrous oxalate is decomposed in hydrogen at 340°C for 2 hr., 36 min and at 360°C for 36 min. When the decomposition proceeds further, Fe, Fe$_3$C and Fe$_3$O$_4$ in bulk are obtained. An analysis of the high temperature Mossbauer spectra gives a value of 5.58 x 10$^{-16}$ ergs/second and 7.026 x 10$^{-16}$ ergs/second for the anisotropy energy of Fe$_3$C obtained through ferrous oxalate decomposition in hydrogen for 1 hr and 2 hr.

Decomposition of mixed metal oxalates of different Fe to Ni ratios yields alloy powders at considerably lower temperatures of 300°C than the conventional methods. Fe$_3$O$_4$ fine particles are confirmed to be present in some
cases of decomposition through low temperature Mossbauer studies. Fe$_3$O$_4$ is an intermediate in the decomposition of mixed metal oxalates pointing to the ferrous oxalate decomposition in the mixed metal. In horizontal experiments, this phase is masked off by the early production of alloy, as the decomposition in horizontal experiment is faster. The FeNi alloy formed from the decomposition of ferrous nickel oxalate, moves from Fe rich to Ni rich side as the Ni concentration in the alloy increases.

The saturation magnetization of the alloy is plotted against the atomic concentration and the curve obtained is compared with the work of Johnson et al (1961) and the Slater-Pauling diagram. The deviation is attributed to the presence of alloys with different compositions in every product of decomposition. This deviation is to the right of Johnson et al (1961) curve. Nickel powder along with FeNi alloy powder is produced. The amount of predominant Ni and FeNi alloy powder separated increases as the Fe content in the mixed metal increases. At high Ni content, it is almost homogeneous.

X-ray studies are used as complementary data to identify the Fe$_3$O$_4$, Fe$_3$C and Fe phases present in the decomposition products of ferrous oxalate in hydrogen and FeNi phase in the decomposition of mixed metal oxalate.
in hydrogen. The scanning electron microscope pictures indicate that morphological changes occur in the decomposing products and at the initial stages of decomposition, the sample is pyramid-like and at later stages of decomposition they agglomerate.