The beamtime awarded has been used for the commissioning of a ReflEXAFS furnace, designed and built in Pavia. In addition, the first experiments have been performed in order to investigate the very early stages of the solid state reaction between Al_2O_3 and NiO to give the $NiAl_2O_4$ spinel.
The experimental protocol was as follows:

1) A thin layer of Ni metal was deposited on a properly oriented Al_2O_3 single crystal, kept in high vacuum at room temperature. The film formation has been monitored with a thickness meter based on a quartz microbalance. We succeeded in depositing films as thin as 50 Å.
2) The Ni layer has been then fired for 20 minutes in pure O_2 (p=0.1 atm) at 700 °C.
3) A further treatment of 6 h in pure O_2 (p=0.1 atm) at 850 °C has been applied.

The following Fig. 1 shows the Ni-K edge ReflEXAFS Fourier transforms of the layer after each of the above three steps. The data have been collected at room temperature.
As it is apparent, after the first stage, only the presence of metallic Ni is detected by ReflEXAFS (see Fig. 1, upper panel).
The second step produces a drastic change in the nature of the film, which now, as it is expected, is composed only by NiO (Fig. 1, middle panel).
Further changes in the ReflEXAFS Fourier transform can be detected after the third step (Fig. 1, lower panel). Now, the ReflEXAFS spectrum is still reminiscent of that of NiO, but it is also clearly apparent, by the drastic decrease in the amplitude of the second shell peak, that the Ni-Ni correlations have been lowered.
Even if more experimental work is needed, we share the opinion that the above experimental finding can be attributed to the formation of a sort of disordered NiO phase at high temperature, which preludes to the real solid state reaction, that in turn requires higher temperatures (> 900 °C) to take place. In addition, we also share the opinion that the above results show that the ReflEXAFS technique can be successfully used to unveil the kinetics and mechanisms of the very early stages of solid state reactions.

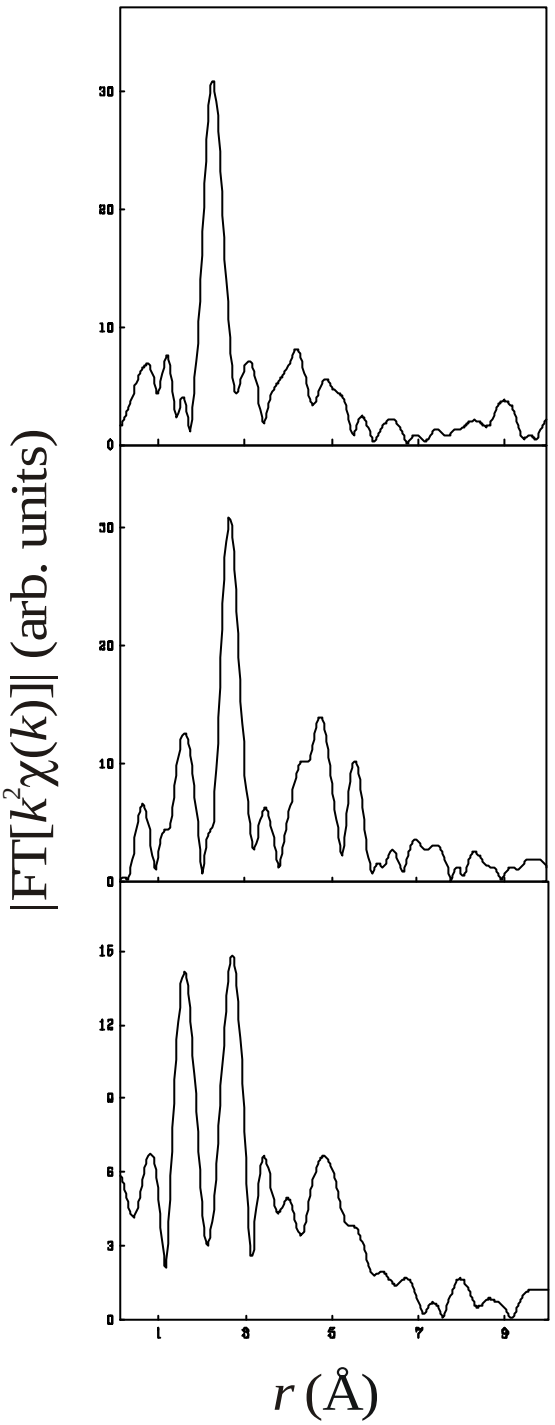

Figure 1.- Ni-K edge ReflEXAFS Fourier transforms of a 50 Å thick Ni layer deposited onto an Al_2O_3 single crystal [(1 0 –1 0) oriented]. Upper panel: Ni layer as deposited; the EXAFS revealed the presence of Ni metal only. Middle panel: after a 20 minutes thermal treatment in pure O_2 (p=0.1 atm) at 700 °C; the formation of NiO is apparent. Lower panel: after a further treatment of 6 h in pure O_2 (p=0.1 atm) at 850 °C; the EXAFS FT is still compatible with that of NiO, but now a dramatic decrease in the amplitude of the second shell peak is apparent.