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Effect of Rolling Reduction on Microstructure and Hardness for Pure Nickel and Pure Iron

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ABSTRACT

Anisotropy as a consequence of cold rolling modes and cold rolling reduction always plays an important role on deformation and mechanical behavior of materials. The present study is aimed at examining the effect of modes and extent of rolling on hardness and microstructure of the pure nickel and pure iron. Hot rolled sheets of pure nickel having thickness of 7 mm and pure iron of thickness 3.2 mm were subjected to different reductions using unidirectional and multi-step cross rolling.

The grains of cold rolled pure nickel and pure iron do not show any significant elongation in ND and TD planes after 30 and 50% reduction in the two experimental modes of rolling such as unidirectional and multi-step. But after giving cold reduction of 85%, the grains of RD planes elongated almost parallel to rolling direction in all the above rolling modes and this elongation is significant in case of unidirectional rolling as compared to multi-step rolling

Keywords:*—AODV, DSDV, Hybrid Routing, Enhanced AODV, ZRP protocols.*

I. INTRODUCTION

For our present research purpose pure iron and nickel metals were chosen because they constitute major part of numerous iron and nickel base alloys [1, 2]. It is known that any metal in its purest form have higher stacking fault energy (SFE) than their alloy forms, which indicates higher and easy deformation characteristics of pure metals with respect to their alloy forms [3]. It is also proven that iron and nickel base alloys show mechanical property anisotropy after cold working, which can be further varied by different mode of cold rolling and changeable percentage cold reduction[4, 5, 6, 7]. The mechanical property anisotropy of metals and alloys are undesirable for engineering structural application whereas for electrical and magnetic application it is desirable, that leads us to understand the basic idea of anisotropy [8, 9, 10]. So it is considered worth of studying the mechanical property anisotropy and micro

Table 1: Directional Dependence of Mechanical and Physical Properties

Pure Metals	Second order elastic stiffness of cubic crystal (GPa)			Young's modulus in direction (GPa)		Magnetic flux density (Tesla)	
	C_{11}	C_{12}	C_{44}	<111>	<100>	c-axis	90° to c axis
Pure Iron	233	135	118	210	170	1.8	1.2
Pure Nickel	252	151	104	200	140	-	-

structural/texture behavior of pure iron and nickel (which constitutes major alloying elements of most of the present engineering alloys) after cold deformation i.e cold rolling [3, 6]. The directional dependence of mechanical and physical properties was reported in table 1. Pure Nickel plays an important role in alloys which have been developed for specific purposes, e.g. for heating elements, stable electrical resistance elements, thermocouples, sparking plug electrodes. Pure Iron has some special properties such as excellent magnetic properties, improved resistance against corrosion and oxidation in comparison to normal steels, good cold forming capability and ideally suitable for welding.

Pure iron is also used in many applications of aviation construction, nuclear technology, the production of magnets (pole cores, yokes and armatures), in automotive construction, as magnetic shielding, as welding rods and fuse wire, as gasket in the chemical and petrochemical industry, power station construction, as anti -corrosion anode and as galvanizing tank including equipment.

II. Experimental Work

Our conventional cold rolling process is unidirectional rolling using two or four high mills. But it was shown that with various other modes of rolling such as clock rolling, two steps cross rolling, multi -step cross rolling, reverse rolling, warm rolling etc. (Figure 1), mechanical, electrical and magnetic properties of metals/alloys can be altered up to considerable extent. It has been observed that mode of rolling and percentage reductions have significant effect on texture evolution and so on anisotropy of properties.

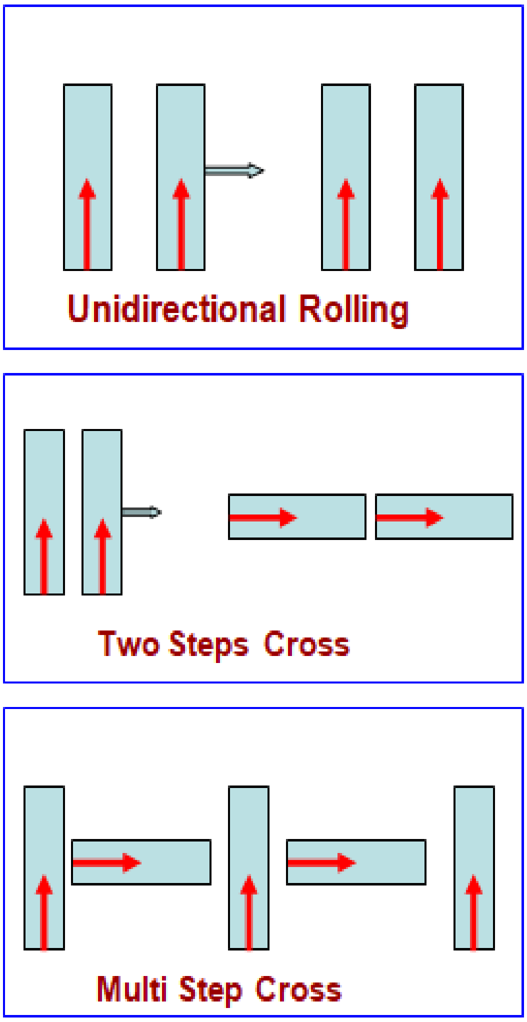


Figure 1: Schematic of different modes of cold rolling.

Table 2: Chemical composition (wt. %) of as received hot rolled pure nickel and pure iron sheets used for present investigation:

Elements	C	Si	Mn	P	S	Cu	Fe	Ni
Pure nickel	0.01	0.01	0.003	-	0.003	0.01	0.20	99.937
Pure iron	0.01	-	0.06	0.003	0.003	0.02	99.904	-

Table 3: No. of samples cold rolled with different modes of rolling and percentage reduction.

As received pure metals in the form of hot rolled sheets.	No of samples unidirectional cold rolled with reduction of			No. of Samples, multi-step cold rolled with reduction of			Total nos. of samples for each metals
	30%	50%	85%	30%	50%	85%	
Pure Nickel	1	1	1	1	1	1	6
Pure Iron	1	1	1	1	1	1	6

2.1 Material

In this investigation, pure nickel and iron with 99.9% purity hot rolled sheets were taken as starting materials and the chemical composition of the metals were given in table 2. Pure nickel and iron were received in hot rolled condition having thickness 7.0 mm and 3.2 mm respectively. Each sample was cut into dimensions of 30 mm (length) X 20 mm (width) X 7.0mm (thickness) for Nickel and samples of dimension 30 mm (length) X 20 mm (width) X 3.2 mm (thickness) were cut from pure Iron for further cold rolling.

Each cold rolled samples in particular rolling mode and for a particular percentage of reduction and for a specific metal were given an unique identification as given below;

Description	% reduction		
	30	50	85
Cold Rolled Nickel Unidi-rectional	CRNU30	CRNU50	CRNU85
Cold Rolled Nickel Multi step	CRNM30	CRNM50	CRNM85
Cold Rolled Iron Unidirec-tional	CRIU30	CRIU50	CRIU85
Cold Rolled Iron Multi step	CRIM30	CRIM50	CRIM85

2.3 Tensile tests

The tensile specimens were cut from the as received hot rolled sheets of pure nickel and pure iron in three different directions namely, longitudinal (L or 0º), 45º (specimen axis at 45º to the rolling direction) and transverse (T or 90º) directions. The longitudinal direction corresponds to the rolling direction (RD), transverse direction (TD) is the direction perpendicular to the rolling direction and ND is specimen axis at 45º to the rolling direction. The schematic diagram of tensile specimen used in present study is shown in Figure 2 as per ASTM standard [11]. These tests were conducted at room temperature at a normal strain rate (10^{-3} s^{-1}) on a screw driven Instron 1185 testing machine. Three specimens were tested for each of the conditions and average values of yield strength (YS), ultimate tensile strength (UTS) and elongation are calculated. From the tensile test values of pure nickel and pure iron in three different directions (0°, 45° and 90° to longitudinal rolling direction) in hot rolled condition, the in-plane anisotropy (A_{IP}) and isotropy Index (d) were calculated to exhibit starting degree of anisotropy of the as received materials.

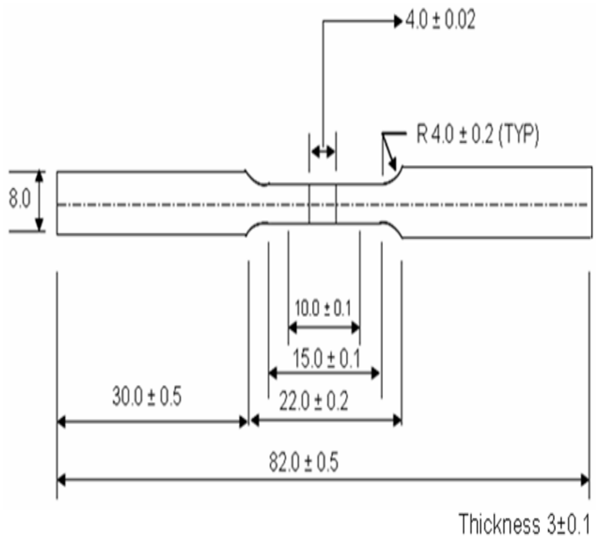


Figure 2: A schematic diagram of tensile specimen showing the sample dimension.

2.4 Metallography

The samples for metallographic examination were prepared through conventional grinding and polishing [12]. The pieces of the specimens were mounted in bakelite mount and polished initially by belt grinder to remove outer rough surface or coating materials. Seven grades namely, 50, 150, 220, 330, 400, 600 and 800 grit polishing SiC papers were used for mechanical polishing. Subsequently, the mechanical polished samples were subjected to selvet cloth polishing with 9µm (coarse cloth polishing), 3µm (medium cloth polishing) and 1/2 µm (fine cloth polishing) diamond paste. The samples were kept in acetone and cleaned by ultrasonic waves for 5 min. The ultrasonic cleaning was repeated two to three times for each specimen. The polished samples were analyzed in etched conditions. The samples of pure nickel were etched with 7% natal solutions whereas the pure iron samples were etched with super picral solution. The etched samples were examined in optical microscope (Leica Microsystems CMS GmbH).

III. Results and Discussion

3.1 Optical microstructure of cold rolled pure nickel and pure iron-

The grain size of as received hot rolled pure iron was found to be 6-8 ASTM No. whereas the coarse grain size of ASTM No.1 for hot rolled pure nickel was observed. The optical microstructure of pure nickel and pure iron, in 30 %, 50 % and 85 % cold rolled conditions have shown in Figure 3 to 6, which show the systematic variation in the microstructure with increase in cold rolling reductions and with variations in rolling modes.

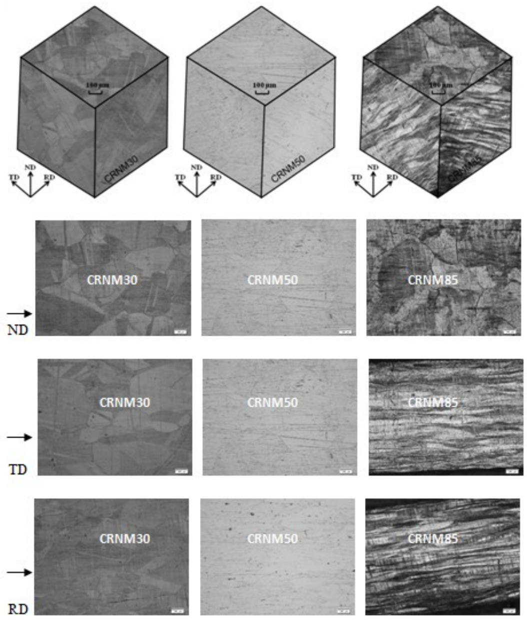


Figure 3: Optical microstructure (3D and 2D view) of multi step cold rolled pure nickel after cold reduction of 30%, 50% and 85%. [Scale @ 100 mm].

The microstructure of 30 % multi step and unidirectional cold rolled (CR) specimens of both pure iron and nickel consists of typical prior equiaxed grains which do not show any significant elongation along the ND and TD planes but exhibits slight stretched grains in the rolling direction, RD (Figure 3 to 6). As the percentage cold reduction increases to 50% the grains of both pure nickel and iron starts showing significant elongation in all ND, TD and RD planes as shown in figures. After giving cold reduction of 85% the grains of RD planes elongated almost parallel to rolling directions. Some of the flow lines are parallel to RD while some are quite away from it. The grains are elongated along RD but it is nearly 8-10º away from the rolling direction in RD plane.

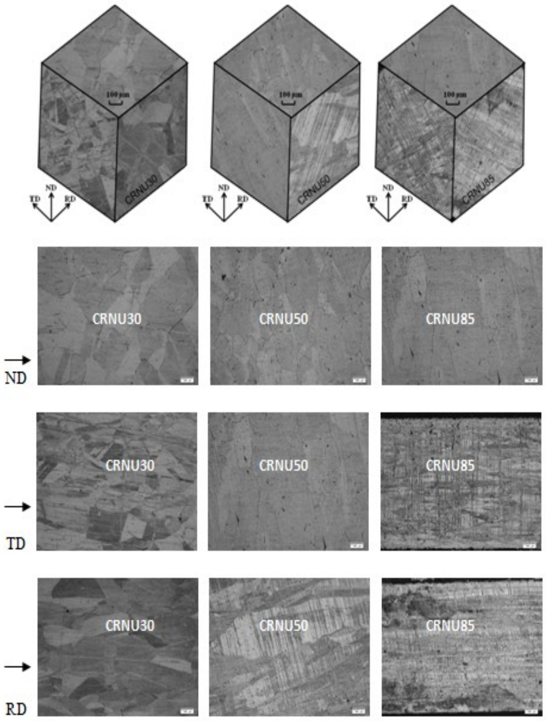


Figure 4: Optical microstructure (3D and 2D view) of unidirectional cold rolled pure nickel after cold reduction of 30%, 50% and 85%. [Scale @ 100 mm].

The grains of pure nickel (starting grain size = ASTM No. 1 and 2) show relatively larger equiaxed size in ND and TD planes after multi step cold reduction of 50 and 85 percent in comparison with the cold reduction of 30% in the same mode. This can be explained on the basis of both lateral and longitudinal elongation during passes of multi-step rolling. But the 85 % unidirectional CR pure nickel specimen shows elongated grains parallel to rolling direction for almost all the planes.

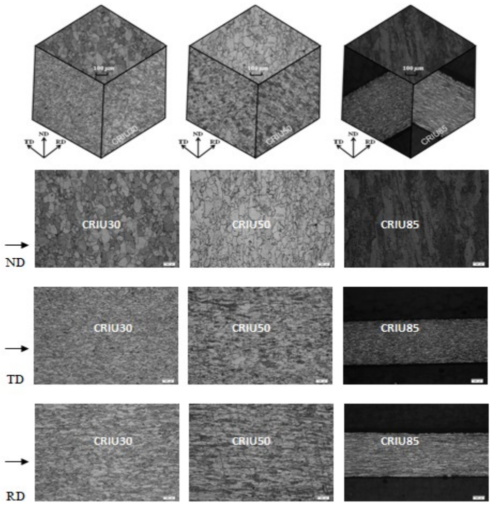


Figure 5: Optical microstructure (3D and 2D view) of multi step cold rolled pure iron after cold reduction of 30%, 50% and 85%. [Scale@100 mm].

The inherent grain size for pure iron is relatively finer (ASTM No. 6 and 8) and so the variation in grain sizes after 30 and 50 percent cold reduction is negligible in ND and TD planes for all the modes of rolling. However after 85 percent cold reduction the grains are elongated along the rolling direction even in the ND planes and the extent of elongation is much higher for unidirectional rolling as compared to multi step and two steps cross rolling (Figure 5 to 6).

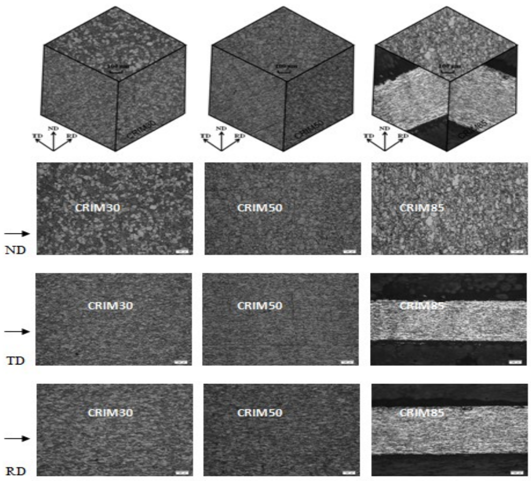


Figure 6: Optical microstructure (3D and 2D view) of unidirectional cold rolled pure iron after cold reduction of 30%, 50% and 85%. [Scale @ 100 mm].

3.2 Tensile properties of as received hot rolled pure iron and nickel-

The average tensile test values of as received hot rolled (HR) pure nickel and pure iron sheets were given in table 4. Tensile test was performed in three directions [0° with respect to the rolling direction (Longitudinal or L), 45° to the rolling direction (45°) and 90° to the rolling direction (Transverse or T)]. Average of three tensile test values tested in all the three directions were reported in table 4. From the tensile test values given in table 4 it can be seen that the yield stress and ultimate tensile stress are highest for specimens with 45° orientation and lowest for longitudinal specimens, for both pure iron and nickel. This indicates that the as received pure nickel and iron have some degree of anisotropy after hot rolling. It can further be noticed that the percentage elongation in 45° orientation is maximum in the case of pure nickel and minimum in the case of pure iron.

Table 4: Average tensile test values of as received hot rolled sheets of pure nickel and pure iron in different orientations.

Orientation of pure metal (HR) specimen's w.r.t longitudinal rolling direction.		0.2% PS MPa	UTS MPa	%El (gauge length of 15 mm)
Longitudinal (0°)	Pure nickel	126	369	58
Transverse (90°)		129	375	56
45°		146	387	59
Longitudinal (0°)	Pure iron	276	326	37
Transverse (90°)		297	332	36
45°		302	340	34

The degree of anisotropy after hot rolling was quantified with two parameters, In-plane anisotropy (A_{IP}) and Anisotropy index (d), for hot rolled pure nickel and pure iron. In-plane anisotropy (A_{IP}) and Anisotropy index (d) were calculated as per formulae in table 5. The formulae used are as follows,

A_{IP} = [2*YS (45°) – YS (T) – YS (L)] X 100 / 2*YS (45°) for both nickel and iron
d = [% El (45°) - % El (T)] X100 / [% El (45°) + % El (T)] for nickel
d = [% El (L) - % El (45°)] X100 / [% El (45°) + % El (L)] for iron

Table 5: In-Plane Anisotropy (A_{IP}) and Anisotropy Index (d)

Anisotropy parameters	In-plane anisotropy (A_{IP})	Anisotropy index (d)
HR pure nickel sheet	12.6712	2.6087
HR pure iron sheet	5.1325	4.2254

The higher values of In-plane anisotropy (A_{IP}) parameter for pure nickel in comparison to pure iron is as expected, which is due to the inherent higher stacking fault energy of pure nickel than pure iron. So it can be noticed that our starting materials, identified as HRNiU (Hot Rolled Nickel Unidirectional sheet) and HRIrU (Hot Rolled Iron Unidirectional sheet), having some degree of anisotropy in mechanical property and the extent of anisotropy is prominent in the case of pure nickel as compared to pure iron due to high staking fault energy.

3.3 Knoop microhardness

The yield surfaces of the materials were determined by Knoop hardness method used by Lee et al. [11], based on Wheeler and Ireland approach [10]. In order to have better statistical average of microhardness, fifteen indentations were taken in each of the six orientations. The average microhardness (KHN) of pure nickel and pure iron in each ND, TD and RD planes for different modes of rolling (unidirectional and multi-step) and percentage reductions (30 %, 50 % and 85

%) are reported in table 6 and 7. The range of micro hardness for pure nickel is from 181 to 276 KHN and the range for pure iron is from 159 to 288 KHN as given in table 6 and 7 respectively.

Table 6: Average Microhardness of Pure Nickel in ND, TD and RD Planes for Different Modes of Rolling and Percentage Reductions

CRNU30	Orienta-tion	KHN	CRNM30	Orien-tation	KHN
	ND (a)	188		ND (a)	188
	ND (b)	184		ND (b)	181
	TD (c)	210		TD (c)	190
	TD (d)	200		TD (d)	194
	RD (e)	207		RD (e)	192
	RD (f)	199		RD (f)	187
CRNU50	**Orienta-tion**	**KHN**	**CRNM50**	**Orien-tation**	**KHN**
	ND (a)	201		ND (a)	199
	ND (b)	235		ND (b)	187
	TD (c)	210		TD (c)	208
	TD (d)	233		TD (d)	184
	RD (e)	227		RD (e)	203
	RD (f)	217		RD (f)	223
CRNU85	**Orienta-tion**	**KHN**	**CRNM85**	**Orien-tation**	**KHN**
	ND (a)	223		ND (a)	260
	ND (b)	244		ND (b)	251
	TD (c)	236		TD (c)	255
	TD (d)	238		TD (d)	248
	RD (e)	262		RD (e)	233
	RD (f)	239		RD (f)	253

It can be seen from figure 7 that the microhardness of TD plane of 30% cold rolled pure nickel is higher than the ND and RD planes for the two modes of rolling. But for the case of 50% reduction hardness of RD plane is relatively more than TD and ND planes. It can further be noticed that hardness of ND plane is always lowest of all planes for the two experimental modes of rolling. With 85% of cold reduction it can be observed that hardness of RD plane is higher than TD plane and hardness of TD plane is more than ND plane for unidirectional rolling mode.

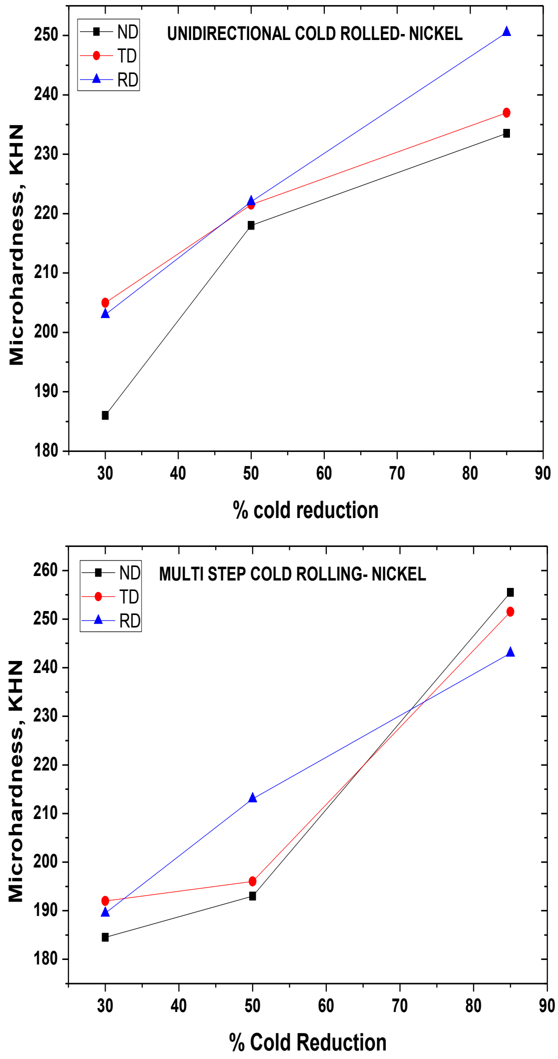


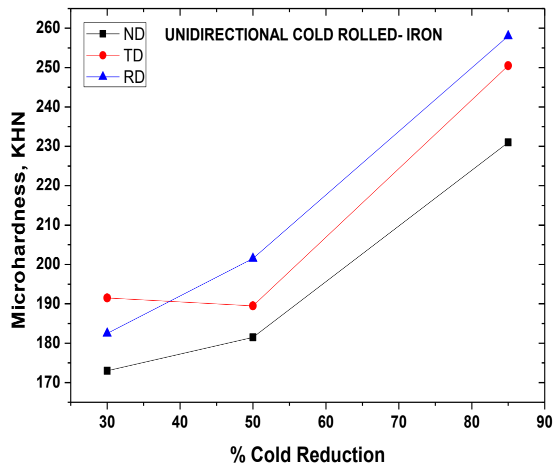
Figure 7: Variation of micro hardness on ND, TD, and RD planes of pure nickel as a function of percentage cold reduction in different modes of cold rolling

In another way of presentation, the variation in micro hardness at particular percentage of reduction in all the two rolling modes as given in table 6. It can be seen that at 30% reduction, the hardness of TD plane is highest for the two modes and decreases from unidirectional mode of rolling to the multi step mode of rolling. Similar trend of decrease in hardness can be seen for RD plane also for same 30% reduction. It can be seen that at 85% reduction the hardness of ND and TD planes increase from unidirectional mode of rolling to the multi step mode of rolling.

Table 7: Average Microhardness of Pure Iron in ND, TD and RD Planes for Different Modes of Rolling and Percentage Reductions

CRIU 30	Orienta-tion	KHN	CRI M30	Orienta-tion	KHN
	ND (a)	159		ND (a)	178
	ND (b)	187		ND (b)	161
	TD (c)	186		TD (c)	177
	TD (d)	197		TD (d)	181
	RD (e)	190		RD (e)	172
	RD (f)	175		RD (f)	164
CRIU 50	**Orienta-tion**	**KHN**	**CRI M50**	**Orienta-tion**	**KHN**
	ND (a)	183		ND (a)	178
	ND (b)	180		ND (b)	181
	TD (c)	203		TD (c)	196
	TD (d)	176		TD (d)	211
	RD (e)	198		RD (e)	212
	RD (f)	205		RD (f)	177
CRIU 85	**Orienta-tion**	**KHN**	**CRI M85**	**Orienta-tion**	**KHN**
	ND (a)	254		ND (a)	226
	ND (b)	208		ND (b)	224
	TD (c)	285		TD (c)	264
	TD (d)	216		TD (d)	246
	RD (e)	228		RD (e)	248
	RD (f)	288		RD (f)	225

In the similar way it can be seen from the figure 8 that the microhardness of TD plane of 30% cold rolled pure iron is higher than the ND and RD planes for all two modes of rolling. But for the case of 50% reduction, hardness of RD plane is more than TD and ND planes only in the case of unidirectional cold rolling. But, in the case of multi-step rolling the hardness of the TD plane is always higher than the ND and RD planes. It can further be noticed that hardness of ND plane is always lowest of all planes for all the two experimental modes of rolling. With 85% of cold reduction it can be observed that hardness of RD plane is higher than TD plane and hardness of TD plane is more than ND plane for unidirectional mode of rolling.



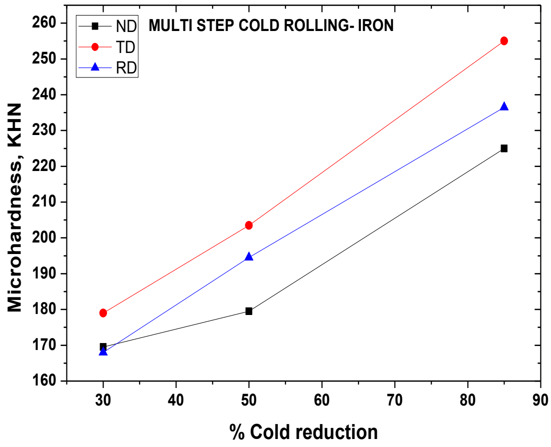


Figure 8: Variation of micro hardness on ND, TD, and RD planes of pure iron as a function of percentage cold reduction in different modes of cold rolling

However with multi-step rolling the hardness of TD plane is higher than both ND and RD planes. In another way of staging the hardness data it can be seen that the micro hardness at particular percentage of reduction varies with modes of rolling modes for pure iron as given in table 7. It can be noticed that the hardness of ND, TD and RD planes, at 30% and 50% reduction, first higher for unidirectional less for multi step rolling mode. After 85% of cold reduction hardness of both TD and RD planes first lower value is observed for unidirectional where as higher value for multi step rolling mode. The trend of change in hardness for ND plane remains same at 85% reduction as was for 30 and 50% cold reduction.

IV. CONCLUSIONS

The In-plane anisotropy (A_{IP}) in respect of tensile property of the hot rolled pure nickel and pure iron shows higher degree of anisotropy for pure nickel as compared to pure iron. The grains of cold rolled pure nickel and pure iron do not show any significant elongation in ND and TD planes after 30 and 50% reduction in the two experimental modes of rolling such as unidirectional and multi-step. But after giving cold reduction of 85%, the grains of RD planes elongated almost parallel to rolling direction in all the above rolling modes and this elongation is significant in case of unidirectional rolling as compared to multi-step rolling. Some of the flow lines are parallel to RD plane while some are quite away from it for all the rolling modes. At 30% cold reduction, TD planes have maximum microhardness values for pure nickel in all the modes of rolling. As the percentage reduction increases the RD planes exhibits maximum hardness value except in the case of 85% multi step cross rolling where it has minimum value of all the planes (here ND plane has highest hardness value).

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