Preparation of Functional Oxide Multilayers

Part 1 – Physical and technical background

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Abstract

In this practical course the goal is to prepare perovskite thin films, either $LaMnO_3$ or $SrMnO_3$. Current research tries to combine properties of different single layers, like ferromagnetism and ferroelectricity, by growing thin films on top of each other and introducing some coupling between the properties of the layers. This is however very time consuming, therefore we just grow a single layer in the lab course. We use nevertheless a state of the art Oxide-MBE system to introduce you to all aspects of Oxide thin film growth. This starts with the preparation of the substrate, the characterization of the substrate with LEED, proceeds with the rate calibration of the sources for different elements, and ends with the in situ characterization of the grown films with RHEED. Finally the samples will be analyzed with X-rays with respect to the film thickness.

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Figure 1: In a ferromagnet the magnetization is switched by applying a magnetic field. In a ferroelectric the charge polarization is switched by applying an electric field. In a multiferroic material both ferroic orders coexist. If there is a magneto electric coupling, the magnetization can be switched (and generally speaking: changed, e.g. reduced) by applying an electric or a magnetic field and the charge polarization can be switched by applying a magnetic or an electric field.

1 Introduction to Multifunctional Oxides

The field of oxides in general covers almost a countless number of different compounds. With the enormous number of compounds comes a great variety of effects like superconductivity, thermoelectricity, the magnetocaloric effect, molecular magnets, magnetoresistivity, ferromagnetism and ferroelectricity, to name a few. These effects originates in the symmetry of the crystals, their electronic correlations, exchange interaction, strain, orbital ordering and so forth (for a comprehensive reading please refer to [1]). Restricting investigations to transition metal oxides only, still provides a large number of compounds. They can be classified in:

- binary oxides: MO, where M stands for a metal ion. MO_2 , e.g. Rutile (TiO-2).
- ternary oxides: ABO₃, or Perovskites. A₂BO₄, called K₂NiF₄ structure. AB₂O-4, spinel structure, e.g MgAl₂O₄.

Here we just focus on the Perovskites, ABO₃. An example of this structure type would be SrTiO₃, the substrate used within this course. Simplified the ABO₃ structure can be considered as cubic. The A ions sit on the corners of the cube, the B ion sits in the center and the Oxygen atoms in the centers of the cube faces. A variant of the ABO₃ perovskites is a substitution of the A ions with other ions, e.g. $La_{1-x}Sr_xMnO_3$. In such a compound the number of electrons contributing to the electronic structure can be adjusted by the x-value. This leads to a great variety of physical properties. By changing the ratio from La to Sr it is possible to change from a ferromagnet to an antiferromagnet or to increase or decrease the Curie temperature.

An example of *multi-functional* oxides are multiferroic materials. These materials are both ferroelectric and ferromagnetic. We assume some kind of interaction between both properties, see Fig. 1.* For reasons of symmetry (see Tab. 1) those materials are scarce and only exhibit a helical or antiferromagnetic order. But on the other hand, ferroelectricity or ferromagnetism alone can be found in many compounds. From this, the idea arises to combine one ferromagnetic layer with a ferroelectric layer and hope for some

^{*}we omit ferroelasticity here

time-reversal			
·	invariant	change sign	
space-inversion			
invariant	ferroelasticity: strain	ferromagnetism: spins	
change sign	ferroelectricity: charge-dipoles	ferrotoroicity: spin-vortex	

Table 1: Symmetries and symmetry breaking within the different ferroic orders. From [2]

interaction at the interface. Assuming such an interaction exists, originating e.g. from strain or orbital ordering, it would be possible to control the magnetic state of one layer by manipulating the charge polarization of the other layer. A possible application would be a non volatile memory device switchable by applying a voltage resulting in a low power consumption compared to conventional switching devices.

2 Introduction to Molecular Beam Epitaxy

Molecular Beam Epitaxy (MBE) is a process, in which one or more materials are heated up and hence are evaporated and finally recondensed on a substrate in crystalline order. Depending on the material substrate combination the crystal grows layer by layer. Layer means one layer of atoms or the building blocks of the crystal, like a perovskite unit cell. This is the fuzzy meaning of a mono layer. Of course, this is a very slow process but it has many advantages. For instance you can develop multilayers of different materials which change from layer to layer. Thus, you can create new crystals, never found in nature, with completely new properties, e.g. a two dimensional electron gas.

Within this section I want to introduce the basic vacuum techniques, which are necessary for MBE. This is followed by some considerations about films and substrates. Then I want to give an overview on the different sources and how to calibrate rates. Finally the oxidation technique is introduced and the most important in-situ characterization methods will be explained. But firstly I want to mention some more thin film techniques.

2.1 Thin Film Techniques

Producing thin films is of importance for industry and research. In industry the techniques have to be fast in order to keep costs low. As MBE is a very slow technique it is almost only found in research. But besides MBE there is a variety of methods applied. This includes sputtering, spin coating, solgel processes, pulsed laser deposition (PLD), and chemical vapour deposition (CVD). As in the field of oxide research not only MBE is applied, the two most common other methods, PLD and Sputtering, are introduced shortly in the following.



Figure 2: The Oxide MBE used in the practical course.

2.1.1 Pulsed Laser Deposition

With PLD different materials are not co-evaporated but a target with the proper stoichiometry is used. This target is heated up locally with short, strong laser pulses. These pulses are mostly in the UV-range and apply pulses with a flux density of a few Jcm^{-2} . As the source is really point like, the inhomogeneity of the layer thickness over the substrate is noticeable in most cases.

2.1.2 Sputtering

The term sputtering refers to a variety of methods. The most common technique is magneton sputtering. A strong magnetic field is placed around a target made of the material you want to evaporate. Additionally some sputter gas is needed, e.g. Ar or O_2 , at low pressures in the range of 10^{-6} to 10^{-3} torr. Applying a high voltage, either ac or dc, depending on the conductivity of the target will ionize the gas and accelerate it towards the target. The collisions of the gas with the target will break out atoms, molecules or clusters which can then move to the substrate.

A special technique used for oxides is high pressure sputtering. Here the sputter gas is O_2 at pressures around one millibar. The substrate is placed in close proximity to the target. The high oxygen pressure minimizes resputtering on the substrate as the mean free path is very short. Additionally the high oxygen pressure minimizes oxygen deficiency.



Figure 3: High oxygen pressure sputtering apparatus: basic parts of HOPSA.

2.2 Vacuum Basics

Good vacuum is the prerequisite for thin film growth with MBE and most of the other techniques. This can be explained by the covering time of a surface. This is the time until a clean surface is covered with one layer by atoms and molecules from the surrounding gases. You cannot grow a clean thin film if your surface is contaminated with "dirt" from the air. Fig. 4 should give you an idea of the values connected with the pressure.

To achieve pressures as low as 1×10^{-11} torr (1 torr = 1.33 mbar, named after Torricelli, who invented the mercury barometer) special techniques are required. Usually a vacuum chamber is pumped with a combination of one pump for the rough to medium vacuum range and a pump for the ultra high vacuum (UHV) range connected in series. The most commonly used pumps today are a combination of a scroll pump with a turbo molecular pump. Wikipedia shows a nice animated GIF illustrating the mechanism of a scroll pump [3]. A turbo pump works with rotors which have to run at a speed comparable to the velocity of the atoms and molecules at ambient temperature. It is a small calculation to show, that for a pump with a rotor diameter of 60 mm 1500 turns per second (!) are just enough. This makes the turbo pumps one of the fastest moving machines. Of course there are many more possibilities available, like cryo pumps or ion getter pumps. Describing them all would go far beyond the goal of this introduction. But, for instance, oerlikon offers a complete guide for free download covering everything on vacuum technology [4].

Measuring low pressures goes far beyond the mercury manometer. For pressures down to 1×10^{-4} torr vacuum gauges based on heat transport are used. They are called Pirani gauges and are based on the pressure dependent heat transport of gases. Pressures between 5×10^{-4} torr and 1×10^{-11} torr are measured based on the ionization of the residual gases. There are two possibilities. First with a **cold** cathode: A voltage of several kV is applied between a cathode and an anode. This ionizes the residual gas and thus a current can be measured, which is dependent on the pressure and the type of gas. Second, the **hot** cathode pressure gauge or Bayard-Alpert gauge: Here the hot cathode (heated by a current going through it) produces electrons which are accelerated to the anode by a small potential. A collector, on the potential of the cathode collects ions which were created by the electrons on their way to the anode. Again, this current relates to the pressure. By tuning the heating current through the cathode the gauge can be tuned for several gas sorts and pressure ranges.



Figure 4: Comparison between the pressure, the particle density, the mean free path, and the vacuum depending on the height within the atmosphere. It is noticeable, that the covering time of a clean surface ranges from a few nano seconds at normal pressure up to several days at 10×10^{-11} mbar which can be derived from the particle density.

2.3 Thin Film Growth with MBE

2.3.1 Preliminary Work – The Substrate

MBE means growing crystals on crystals as the substrates you use are single crystals. The surface of the substrate dictates the arrangement of the adsorbed atoms. Like you cannot arrange cargo containers on a chessboard the lattice spacing of the substrate has to be equal to the lattice spacing of the crystal to be grown. A small lattice mismatch (a few %) can lead to strain in the grown layer which will decrease with the thickness or the layer will have misfit dislocations or both. Even if the lattice constants match it is not guaranteed that the layer will grow. The surface enthalpy has to be right as well. Thus, the right substrate has to be found. Cleaning the surface of the substrate is the next step. As the substrates have to be brought into the system contamination with air and possible oxidation of the surface is unavoidable. Several methods can be applied. One simple method applicable for most oxides is to heat the substrate. You either just evaporate the dirt or in cases of metals (W) the covering oxide layer will be split off due to the lattice mismatch. Another method would be to use Ar-ion sputtering. The fast Ar-ions from a sputter gun bombard the surface and ablate the top layers. They are sputtered off. To smooth the

2.3.2 Sources

There exists a great variety of sources. They differ mostly in the way of the heating mechanism. A very simple approach, used for Ti evaporation, is driving a current through a Ti rod. But most sources are more sophisticated. In Fig. 5 common sources are explained.

One problem is to find a proper material for the crucible to use for a certain material. The crucible material should have a high melting point and should not react or alloy with the material inside, not



Figure 5: Different type of sources in MBE: left *e*-gun: either the crucible containing the material or the material within a crucible is heated by an electron beam. middle radiation heating of a crucible. The surrounding filament is heating the crucible due to heat radiation. right resistive heating of a crucible.

even at high temperatures. When filling La into Al_2O_3 , a crucible material for most other materials, one will only find Al on the substrate. The La takes the Oxygen and the Al will desorb from the crucible. An unhappy situation. In Tab. 2 a few combinations are listed.

To improve homogeneity of the film thickness over a wide range of the sample surface several measures can be taken. Important is the interplay of distance between sample and source and size of the source. If you have a point like source, the evaporated material will leave it like a spherical wave. Increasing the distance now would improve the homogeneity. On the other hand a wide spread source can be considered as a plain wave for small distances. However both solution are not optimal. The point like source is very ineffective while the wide spread source mounted close to the sample, hinders one to mount more sources with different materials. A good solution is a crucible as shown in Fig. 6. Another solution is rotating the sample or move it in any other way around over the sources.

material	source	crucible	melting point	state	density
			[°C]		$[\text{gcm}^{-3}]$
Al	all	W, BN, CG	660	melts	2.7
Ti	e-gun	Ta, CG	1700	melts	4.5
Mn	all	Alumina	1200	sublimates	7.44
Sr	\mathbf{EC}	CG, PBN	1500	semi-melts	2.63
Ba	all	Mo	700	melts	3.65
La	EC	Alumina	900	melts	6.16

Table 2: Different materials for typical oxides and the way they are evaporated. EC stands for effusion cell, CG for coated graphite, PBN for pyrolitic boron nitride.



Figure 6: A long crucible with small opening angle α is very effective in material consumption. The distance to the source can be large. The evaporation characteristic will be a flattened spherical wave limited to the sector given by the opening angle α . Very close to the crucible and in the far field the characteristic can be considered flat. Usually the aspect ratio of crucibles is about four.

2.3.3 Rate Calibration

Rates are **the** parameter determining whether a film will grow epitaxially or amorphous. Thus the determination of the growth rates is of utmost importance. A very simple way of measuring the growth rate is to use a so called quartz micro balance (QMB) which is mounted close to the sample position. Such a QMB consists of a small flat quartz crystal, its resonant frequency depending on the mass of the crystal. If material is deposited on the surface of the quartz, the mass is increased which leads to a decreased resonance frequency. A simple formula holds for changes of around 15%:

$$\frac{\Delta m}{m} \propto -\frac{\Delta \nu}{\nu}.\tag{1}$$

Here *m* is the mass of the crystal, ν the resonance frequency. The Δ 's describe the change due to the deposition of material on the surface. As frequencies can be measured with high accuracy (0.01 Hz resolution at a base frequency of 6 MHz can be achieved without *advanced magic*), very small changes in mass can be determined (10⁻⁸). An example would be iron, which leads to a frequency change of around 8 Hz per monolayer. Thus, sub Å resolution can be accomplished.

To calibrate rates with a QMB the easiest approach is to monitor the change in frequency during film growth and determine the thickness of the film with an alternative method. But from Eqn. (1) a more useful equation can be derived:

$$\Delta \nu = C \rho d \text{ with } C = \frac{A}{A_{\rm s}} \frac{\nu_{\rm res}^2}{N \rho_{\rm q}}.$$
(2)

d is the layer thickness, ρ and ρ_{q} are the densities of the layer and the quartz, and C is a constant depending on the geometry of the QMB. That is, A is the total area of the crystal, A_{s} the area of the surface covered with the layer, ν_{res} the resonance frequency and N is a constant representing the dependence of ν_{res} from the thickness of the quartz. With this formula one can estimate the layer thickness before resorting to alternative methods. But, N is widely unknown and the position of the quartz in respect to the sample and source, the so called tooling factor, giving the ratio between the material reaching the quartz and the sample, can only be roughly estimated. Thus, with two unknown factors in the equation an alternative thickness measurement is still necessary for the rate calibration, but this time the thickness can be estimated beforehand.

2.3.4 Oxidation

Oxidation is usually achieved by supplying atomic Oxygen during the film growth to the sample. This can be done with an Ozone generator or an atom source based on a small plasma chamber. The amount

of Oxygen delivered to the sample can be measured indirectly using the partial pressure or directly using a mass flow controller. The flow determines the rates used for the film growth. The rate of the material to be oxidized should be adjusted in a way, that the oxidation is complete at the substrate but no oxidation takes place at the material within the evaporation source. This working point can be determined by slowly increasing the Oxygen flow at a constant evaporation rate of the material, and monitoring the displayed rate on the QMB. With increasing flow the rate displayed by the OMB should increase until all atoms are oxidized. Then a plateau is observed, followed by a decreasing rate, as now the material within the source starts to oxidize. This working point has to be found for each desired growth rate of the material.

2.3.5 Growth Process

For the growth of a complex oxide two methods are applied. The first method is the co-deposition. When using co-deposition the rates of every constituent is set according to the desired stoichiometry and the oxygen is supplied in the same way as described in the previous section. Then the shutters are opened simultaneously. Thus, all the atoms reach the substrate and build the layer.

The second method is called shuttered growth and requires a RHEED setup in the growth chamber (see Sec. 2.4.3). Then, the layer is grown in half layers of the two oxide constituents. That is, a ABO₃ is grown in two steps: first the AO, then the BO₂. The successful growth of a half layer is hereby monitored with the RHEED. For the growth, the oxygen flow is set like that described above. Then the shutter for one material is opened until the RHEED shows that the first half layer is finished. The shutter is closed again. The second half layer follows in the same way.

2.4 In-Situ Characterization Methods

The Oxide MBE is equipped with three in-situ characterization possibilities. That is, Auger Electron Spectroscpy - (AES), Low Energy Electron Diffraction - (LEED), and Reflection High Energy Electron Diffraction - (RHEED). With an AES the chemical composition of the surface can be determined. LEED and RHEED are electron scattering methods sensitive to the crystalline quality of the surface. For the two latter methods I recommend reading the scattering basics, Sec 3.1 first.

2.4.1 Auger Electron Spectroscopy - AES

The basis of the AES is a bombardment of the atoms within the sample with high energy electrons. Thus, electrons of the inner shells are removed from the atom.

If an atom has an inner shell electron removed, it usually relaxes to the ground state by filling the hole with electrons from the outer shells and emitting X-ray photons. An alternative process for returning to the ground state is the Auger process (Fig.7, right). Here electrons move to free states in the inner shells without emitting photons, but transfer their energy to other electrons. These can leave the atom with a characteristic kinetic energy. The probability of the Auger process decreases with the atomic number of the atom. The efficiency for X-ray emission goes opposite (Fig. 7, left).

The Auger process starts by taking an electron from the K shell. An electron from the L-shell *falls* down to the K shell filling the hole there. The energy gain from this transition is directly transferred to another electron of the L shell, which now can escape the atom. Now the L shell has two electrons missing. These holes are filled with electrons from higher shells (M, N, ...). This process again is accompanied by photon emission or energy transfer to other electrons. As the states have characteristic energies, the transitions and thus the energy transfer is characteristic for the atom sort as well. If the secondary



Figure 7: Left: The yield of X-ray emission upon excitation of an atom depending on the atomic number Z. Right: Schematic of the Auger process. 1) A high energy electron hits the atom and takes out an electron from the K shell. 2) An electron relaxes from the L shell, transferring its energy to an Auger electron. Based loosely on [5].



Figure 8: Typical Auger spectrum of Ti and Mn: Plotted is the derivative of the intensity (or the electron count) in respect to the energy versus the electron kinetic energy. Based on [6].

electrons from a sample, after bombarding it with high energy electrons, are carefully analyzed with respect to their kinetic energy, a spectrum is obtained (Fig. 8). This spectrum is unique for each element.

The AES itself consists of an electron gun, an energy analyzer, and an electron detector. The electron gun delivers electrons, typically with 3 keV kinetic energy to the sample. In backscattering geometry the analyzer is tuned through the energy range of the Auger electrons and the detector records the relative intensities. In this way the Auger spectrum is obtained.

As the mean free path of electrons with energies typical for the Auger process is in the range of tenth of nanometers, see Fig 10, the method is very surface sensitive.



Figure 9: Sketch of the AES geometry.



Figure 11: left: Sketch of a LEED setup. right: LEED image of LMO at 130 eV. In the middle the electron gun obscures the screen.

2.4.2 Low Energy Electron Diffraction - LEED

LEED is a very simple approach to determine the crystal structure of surfaces. It is basically scattering of electrons on the crystal surface in backscattering geometry. An electron gun supplies electrons with well defined energies in the range of 50 eV to 300 eV. These are backscattered at the surface and mapped on a screen. As the mean free path of the electrons is very small, see Fig 10, the picture on the screen is an image of the reciprocal space of the crystal surface.

2.4.3 Reflection High Energy Electron Diffraction - RHEED

RHEED is quite analog to LEED, but the scattering takes place at grazing incidence. The electron energy is higher than with LEED, namely around 15 keV, but due to the small angle of incidence the method is sensitive to the surface only. A sketch of the Ewald construction in the case of RHEED is shown in Fig. 13. The intensity of the reflections is very sensitive to the smoothness of the surface. A stepped surface leads to 3D scattering and intensity within the rods on the screen is lost. This can be used to observe the growth of single layers. An unfinished layer has a stepped surface e.g. due to island growth. Thus the intensity is low. Once the the layer is finished it has a 2D surface again. Thus the intensity within the scattering rod is high. This variation in the intensity during the growth is called RHEED



Figure 12: Sketch of the RHEED geometry.



Figure 13: The Ewald sphere in RHEED geometry. A 2D surface of points has rods in reciprocal space. Either the rods have a finite diameter (yellow) or the Ewald sphere has, due to the energy spread of the incoming electrons, a certain spread in the radius. In either way the intersection of the rods with the sphere is smeared out at small angles (red). This leads to lines on the screen.

oscillations. It is only observed when the growth is really layer by layer.

3 Short Introduction to Scattering

Of course the title promises more than the section can hold. The aim of this section is just to explain, why the results with an X-ray reflectometer or LEED look like they do and to give you the basic formulas you need to understand the results when probing thin films.

3.1 Scattering Basics

In the simplest cases of scattering a lot of approximations are used. You consider the source far away from the sample. Thus, you only deal with plane waves at the sample. Further you consider your detector being far away from the sample. Again, you just have to deal with plane waves at the detector, even though you treat the scattering at the sample as the origin of spherical waves. Moreover you assume a



Figure 14: left: scattering geometry for X-ray reflectometry. With roughness (lower panel) the intensity is scattered away from the maxima after Bragg's law. right: scattering geometry in the case of LEED. Here the zero order reflection (n = 1) is back scattered into the source and thus not visible. The first peaks visible on the screen will be of first and higher order $(n \ge 1)$. The smaller circles represent the lowered amount of intensity due to scattering at deeper layers due to the small mean free path of the electrons.

monochromatic source and a wavelength comparable or smaller to the distances relevant for scattering, e.g. the inter atomic distance or the layer thickness. In summary we treat the scattering experiment in the Frauenhofer approximation. To make things even simpler we consider purely elastic scattering. With this we can still treat LEED, RHEED and X-Ray reflectometry, but of course we lost the AES. Now we need some scattering particles, electrons, photons, neutrons and so fourth and some object to scatter at. Describing the scattering particles with waves one fundamental value is of course the de-Broglie wavelength of particles,

$$\lambda_{\rm de-Broglie} = \frac{h}{p} = \frac{h}{\sqrt{2m_{\rm e}E_{\rm kin}}}.$$
(3)

With electrons the kinetic energy is easily determined, as it corresponds to the acceleration voltage, $E_{\rm kin} = eU$. The object to scatter at, our sample, is assumed to be a periodic arrangement of ... ? Ok, here we have to distinguish what we are doing but in total the theory goes the same way. But if we do X-ray reflectometry, the periodic arrangement will, more or less, consist of thin film layers and we just consider the boundaries between these layers for scattering. For LEED and RHEED the electronic shells of the atoms close to the surface build the periodic arrangement we scatter at. In both cases the Bragg equation can be used to calculate the angles of maximum intensity of the scatted waves:

$$2d\sin\theta = n\lambda.\tag{4}$$

In Fig. 14 the geometry and the values are explained. There you also see, that you lose intensity if you have rough surfaces. With the reflection and transmission taken into account Eqn. (4) can be expanded for a multilayer. In the case of LEED the geometry is different but the formula is almost the same:

$$d\cos\theta = n\lambda \text{ or } d\sin\alpha = n\lambda.$$
(5)

As the coherent radius for an electron beam with energies typical for LEED is of the order of 100 Å this can be applied nicely for atoms arranged in a crystal.



Figure 15: Simulation of a reflectometry measurement of a multilayer Fe/Cr-system.

One simple approach to describe all kinds of scattering is based on the Fourier transformation. The intensity distribution is the (2D / 3D) Fourier transformation of the scattering object. Where the reciprocal space of a periodic structure is the Fourier transformation of the real space object. Thus, the scattering intensity distribution maps the reciprocal space. If you take a 2D surface of periodically aligned spots at distance d, like atoms on a crystal surface, the Fourier transformation will lead to periodically aligned rods (RHEED, see Fig. 13). The distances of these rods will be $2\pi/d$. This is pretty analog to optical diffraction at slits and meshes.

3.2 Thin Films measured with small angle X-ray Scattering

We now might be able to understand the general outline of a X-ray reflectometry curve like shown in Fig. 15. The y-axis is plotted in terms of Q which is

$$Q = \frac{4\pi}{\lambda}\sin\theta.$$
 (6)

For Q below 0.06 Å one observes the total reflection plateau, which is not flat, as the beam has a certain diameter and hits the sample holder for really small angles. Then the intensity goes down, the reflectivity is reduced with increasing angles. The larger the angle, the deeper the probe can go into the sample. If the sample is rough, the intensity will drop more rapidly. For Q above 0.1 Å oscillations are visible. These are the maxima from the Bragg equation. The peaks with the small period correspond to the total layer thickness, while the larger peaks with a longer period correspond to the single bilayer thickness.

This was just a very short introduction with the purpose of giving you the basic ideas. Of course scattering is a very wide field from a methods point of view as well as from the sources. Just consider the imaging methods using X-rays in medicine and the huge variety a synchrotron offers. The former IFF published a hand full of books dedicated to scattering like [8], [9], or [10]. In [8] you will find further reading on scattering methods. For the application of X-rays on thin films I want to emphasize Chapter D4: Thin Film Systems: Scattering under Gracing Incidence, especially sections 1 to 5. To see, what else is possible and where the field of scattering is going to I would recommend [9].

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